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NATIONAL UNIVERSITY OF SCIENCE AND TECHNOLOGY POLITEHNICA BUCHAREST

Faculty of Biotechnical Systems Engineering

Department of Biotechnical Systems

Summary of the Doctoral Thesis

Contribuții și cercetări privind procesul de lucru al mașinilor de tratat semințe prin vacuumare

Contributions and researches regarding the working process of vacuum seed treatment machines

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Rezumat

Tratarea semințelor cu insectofungicide în scopul protejării plantelor în primele stadii vegetative este o practică agronomică consemnata încă din antichitate. Pe parcursul istoriei, tratarea semințelor înainte de semănat a beneficiat de două direcții principale de dezvoltare.

Prima abordare vizează dezvoltarea produselor cu care se face tratare, iar cea de-a doua, inovarea echipamentelor utilizate la omogenizarea produsului cu semințele. Partea de cercetare-dezvoltare privitoare la produsele de tratat semințele a atins potențialul maxim odată cu apariția insecticidelor neonicotinoide. Însă, datorita politicilor de protecție a mediului, se recomandă ca până în anul 2030, cantitatea de pesticide utilizate pentru protecția culturilor agricole să fie redusă la jumătate. Pentru a răspunde acestei provocări, tema de cercetare abordată își propune să contribuie cu o noua metodă de tratare a semințelor prin utilizarea vacuumului ca element de ajutor pentru a facilita transferul de pesticide lichide în interiorul semințelor. Pesticidele transferate în structura poroasă a semințelor sunt antrenate mai ușor în sistemul circulator al plantei si expuse radiației solare unde sunt metabolizate în întregime, reducând astfel riscul de contaminare a mediului. Activitățile de cercetare teoretică si experimentală au urmărit tot procesul de dezvoltare a unei tehnologii, începând cu studiul bibliografic, conceperea, proiectarea si realizarea echipamentului de tratare, efectuarea simulărilor numerice pe baza modelelor fizico-matematice si validarea acestora prin experimente de laborator. Prin utilizarea echipamentului de tratat semințele folosind vacuumare, s-a constatat că procesul de lucru al echipamentului nu afectează semnificativ structura morfologică si fiziologia semințelor concomitent cu acoperirea unui spectru controlabil de doze de pesticide asemănător tratamentelor prin omogenizare mecanică în incinte cu tamburi rotativi sau discuri centrifugale.

Abstract

Treating seeds with insectofungicides to protect plants in the early vegetative stages is an agronomic practice recorded since ancient times. Throughout history, pre-sowing seed treatment has seen two main directions of development.

The first approach focuses on the development of the products used for treatment, while the second one involves the innovation of the equipment used for homogenizing the product with the seeds. Research and development related to seed treatment products reached its maximum potential with the advent of neonicotinoid insecticides. However, due to environmental protection policies, it is recommended that by the year 2030, the quantity of pesticides used for the protection of agricultural crops be halved. To address this challenge, the research topic aims to contribute with a new method of seed treatment using vacuum as an aid to facilitate the transfer of liquid pesticides inside the seeds. Pesticides transferred into the porous structure of the seeds are more easily entrained in the plant's circulatory system and exposed to sunlight, where they are fully metabolized, thus reducing the risk of environmental contamination. The theoretical and experimental research activities have followed the entire process of developing a technology, starting with literature review, conceptualization, design, and implementation of the treatment equipment, conducting numerical simulations based on physico-mathematical models, and validating them through laboratory experiments. By using the seed treatment equipment with vacuum, it was observed that the equipment's working process does not significantly affect the morphological and physiological structure of the seeds. Simultaneously, it covers a controllable spectrum of pesticide doses similar to treatments through mechanical homogenization in enclosures with rotating drums or centrifugal discs.

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Gratefulness

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Bucharest, October 2023 *Viorel Fătu*

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Foreword

Cereals for grains are cultivated on an area of over five million hectares in Romania, producing nearly nineteen million tons of seeds according to statistical data published in 2022 by the National Institute of Statistics.

Seed treatment before sowing is an important technological process, as the success of initiating crops in the early vegetative stages depends on the quality of treated seeds. From an ecological perspective, seed treatment has the lowest impact on the environment because the smallest amount of pesticide per hectare is used (for example, 90 grams in seed treatment instead of 140 grams in vegetation treatment with imidacloprid).

From the analysis of the working process of seed treatment equipment, it was found that the kinetic energy of homogenization is distributed to both seeds and the treatment product. On average, the power absorbed by homogenization equipment was around 1 Wh per kilogram of seeds.

The experimental study of the sorption phenomenon of liquid pesticides highlighted that during seed treatment using vacuum, absorption is the main form through which mass transfer occurs.

The doctoral thesis "*Contributions and Research on the Working Process of Seed Treatment Machines through Vacuuming*" is structured into 8 chapters, developed over 181 pages, including 102 figures and charts, 33 tables, mathematical relationships, as well as a bibliography list consisting of 134 references. This work also includes a list of notations and symbols (4 pages), and, in the end, a series of annexes (14 pages) presenting materials and informative data in the form of tables, charts, or figures related to the studies and research presented in this work.

This work is a synthesis of theoretical and experimental research regarding the preparation of seed material before being introduced into the soil, focusing on treatments performed to prevent seeds from being attacked by pests that could compromise germination and affect the subsequent development of plants.

The main objective of the theoretical and experimental research was to study the possibility of reducing the quantity of pesticides used for crop protection by developing new procedures for seed treatment before sowing. The primary goal of the research theme was to identify and describe the physical and biological phenomena that occur during the treatment of seeds using vacuum, as a method to enhance the diffusion of active substances into the porous matrix of the seeds. Secondary objectives included defining the operating parameters of the seed treatment equipment through physical-mathematical simulations and laboratory experiments, among others.

In Chapter 1 of this work, "The Importance of the Topic and the Objectives of the Doctoral Thesis," the significance of the addressed topic is presented, along with the main objective and subsidiary objectives pursued in developing the thesis. The use of seed treatment equipment, vegetation, or soil determines the application method of pesticide substances (insecticides, fungicides, pesticides, rodenticides, acaricides, bactericides, nematicides, molluscicides, avicides, etc.) uniformly, with effective dosage and low aerosol diffusion into the atmosphere, without affecting the basic characteristics of agricultural soil or the environment. Thus, establishing the constructive and functional parameters of vacuum seed treatment equipment represents theoretical and practical challenges on which the quality of the treatment performed and the corresponding reduction of applied substances depend.

Chapter 2, titled "*Aspects regarding the physical properties of seeds and substances for their treatment*", contains some general notions about the process of seed treatment before sowing, the purpose and importance of seed treatment, basic characteristics of seeds (morphological structure, seed physiology, physico-mechanical and technological properties, as well as the basic properties of solutions and substances used in treatments), and parameters involved in the treatment process. Results obtained by researchers in the field are presented, concluding that seed treatment before sowing is a crucial technological process since the success of initiating crops in the early vegetative stages depends on the quality of treated seeds. The performance of seed treatment equally depends on the effectiveness of the product used and the quality of the seed treatment process.

In Chapter 3, entitled *"Methods, Techniques, and Equipment for Seed Treatment"*, methods of treatment are presented based on the aggregate state of applied substances, factors influencing the seed treatment process (biological, physico-chemical, atmospheric, constructive, or process factors), and several methods and technologies used in seed treatment before sowing (dry treatments, wet treatments, and other categories such as fumigation, heat treatment, UV radiation treatment, plasma treatment, etc.). Finally, the seed treatment method using the vacuum phenomenon is described, along with the equipment and apparatus used in seed treatment before sowing, quality requirements and indicators, and the description of their composition and working process. Modern trends in the construction and operation of seed treatment equipment are also presented.

As a conclusion of this chapter, it is stated that the proposed method of seed treatment using vacuum in this thesis presents negligible kinetics of seeds and can borrow characteristics from soaking treatment methods, cold plasma treatment, and treatment with acoustic or sonic pressure pulsations.

Chapter 4, titled *"Current State of Theoretical and Experimental Research on Seed Treatment Equipment",* presents the importance of seed treatments before sowing, a synthesis of theoretical research on the seed treatment process and its mathematical modeling, main elements of simulating (FEM or DEM) the process, and a synthesis of experimental research related to the seed treatment process. The influence of constructive and functional parameters of specific equipment on seed treatments, material balances, and energy balances in seed treatments are also presented. This chapter concludes with some conclusions regarding the importance of understanding the factors influencing the seed treatment process and the main aspects resulting from the analysis of theoretical and experimental research on the treatment process and the level of germination of treated seeds.

Chapter 5, entitled *"Theoretical aspects and contributions regarding the working process of seed treatment equipment before sowing"* presents aspects of sorption and desorption phenomena in the seeds of agricultural crops and influencing factors, as well as some deterministic mathematical models expressing the physical connection between process parameters or relationships regarding the simulation of these processes (sorption-desorption). Additionally, some aspects of the physical-mathematical simulation of the vacuum treatment process of corn seeds are presented, covering a range of treatment duration values of 6 minutes with a sampling rate of 10 seconds and 27 pressure values in the treatment chamber. Using such simulations, the treatment duration and working pressure in the treatment chamber can be established depending on the desired loading percentage of the equipment's technological volume.The chapter concludes with conclusions regarding the applicability and use of these mathematical models, showing that the duration of maintaining seeds in immersion after air extraction from their pores and the pressure at which the vacuuming was performed are the main parameters that need to be controlled in the vacuum treatment process.

Chapter 6, titled *"Experimental research on the seed treatment process before sowing"* encompasses the objectives of experimental research, the material subjected to experimental studies, and its initial characteristics. It includes the numerical modeling of the adsorption of the active solution (mesurol solution), as well as the equipment and tools used for experimental research on the sorption phenomenon in a vacuum and the influence of equipment parameters on the seed treatment process. This involves the technological volume loading with pesticides, or the impact of temperature on pressure and vacuuming time.

Additionally, some experimental studies regarding the dose of the active substance used in seed treatment and the variables of the model determining the quantity of pesticide necessary for the treatment are presented. Experimental testing variations of the active substance at different doses on corn seeds (treatment with imidacloprid) and the results of these studies are also detailed.

Furthermore, experimental research has focused on mass transfer in the process of vacuum seed treatment and establishing the material balance in the process using laboratory equipment for vacuum treatments with pesticide application. It also delves into the energetic parameters of the seed treatment process, specifically the energy balance in vacuum seed treatment (electric power consumption), and the impact of vacuum treatments on plants and the environment.

Conclusions drawn highlight that the analysis of germination and root growth of corn seeds treated with imidacloprid through vacuuming, across a range of doses from 0.25 to 6 kg of active substance, demonstrated that vacuum treatment does not negatively impact seed health, but rather, it is influenced by the concentration of the substance used. Moreover, by using vacuum as a means to accelerate the diffusion of treatment solutions within seeds, a practical spectrum of doses between 0.5 to 5 kg of substance per ton of seeds and a theoretical range between 1 to 14 kg of substance per ton of seeds have been covered. Calculations have also shown a practical energy consumption of less than 1 Wh for treating one kilogram of seeds, with a vacuum pump efficiency of 67%.

In *Chapter 7*, titled *"Numerical and experimental analysis of the seed degassing system in a vacuum"* it is asserted that physical treatments are challenging to use due to a fine line between effectively eradicating diseases transmitted by seeds and injuring the seeds. Not all seed lots react the same way to all treatments, making it difficult to predict how physical treatments will affect seed germination and vigor.

In this context, an analysis of the seed degassing process in a vacuum chamber is conducted, establishing the gas load and final pressure in the degassing process, i.e., the removal of air or other gases from around the seeds or products. A numerical analysis model with finite element components is developed for the experimental model structure used in the thesis. This includes a structural dynamic analysis with finite elements (FEA), covering the vacuum system (vessel, vacuum pump, thermal system), evaluating the pumping time for evacuating a container in the raw or medium and high vacuum region. It also includes transient thermal analysis with finite elements and the method of applying temperature loads throughout the gas load domain.

The chapter concludes with a section of conclusions regarding the results of analytical and experimental research.

In *Chapter 8*, titled "*Final Conclusions. Personal contributions. Recommendations and research perspectives",* final conclusions related to the theoretical and experimental research presented in the thesis are presented. The author synthetically outlines personal contributions and suggests future research directions in the field. Considering the information provided, it is appreciated that the doctoral thesis's topic is contemporary and addresses an essential field, that of applying treatment solutions to seeds before sowing, using the vacuuming method to fill the air gaps in the internal structure of seeds with the treatment substance.

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1. The current situation of pesticide use

Cultivating plants for food represents perhaps the oldest activity of human civilization and is characterized as the main driver of demographic development. Throughout history, plant cultivation activities have always been accompanied by obstacles arising from water scarcity and the attack of living organisms competing for resources or parasitizing cultivated plants [1]. In modern times, these obstacles have been defined as forms of stress that hinder plants from reaching their maximum genetic and phenotypic potential.

The initial measures to control populations of harmful organisms involved mechanical processes such as plowing and hoeing, performed with tools or equipment designed to ease the physical effort exerted by humans. These processes helped control weed populations, negative microbial activity in the soil, and the interruption of certain phenological development processes in the biological cycle of harmful insects but came with a high energy cost.

With the development of knowledge in the field of chemistry, chemical substances began to be studied and used as a means of controlling diseases, pests, and weeds. Chemical treatments for plant protection are classified as foliar treatments, soil treatments, and seed treatments. Among these, seed treatments have the lowest short-term environmental impact, as they use the smallest amount of active substance distributed over the agricultural surface.

The principle of protecting seeds and germinated plants against harmful organisms involves the controlled poisoning of seeds with various substances with biocidal or biostatic properties for the target organisms. During the plant's growth, these substances dilute within the plant's organs to a concentration that poses no risk to human and animal health.

In Romania, cereal crops cover an area of over five million hectares and produce almost nineteen million tons of seeds according to statistical data published in 2022 by the National Institute of Statistics (INS, 2022). Over the last 20 years, the average quantity of pesticides used per hectare has been below 1 kg (FAOSTAT). Considering Romania's average agricultural area of eight million hectares, an annual pesticide consumption of 5.6 million kilograms can be estimated. The average pesticide consumption in the country of 0.7 kg/ha is moderate compared to countries like China or Brazil, where consumption can reach 10-15 kg/ha. This pesticide consumption has been sufficient for crop protection against diseases and pests until now. However, due to climate anomalies observed in recent years, the increasing number and frequency of harmful organism species, and ecological trends to reduce pesticide quantities, agricultural crops are at risk of significant damage without alternative protection measures.

The quantity of insecticides needed for treating seeds intended for establishing grain crops is approximately 450 tons of active substance. By distributing this quantity over the agricultural surface cultivated with grain crops, the average consumption is around 90 g/ha. This quantity is supplemented by about 20-25 grams of fungicides, resulting in an approximate consumption of 115 grams of pesticides per hectare.

Pesticides deposited on the surface of seeds are carried into the plant's circulatory system with water, through diffusion, but some pesticides migrate from the seed surface into the soil depending on water mobility. The amount of migrated pesticides, if not captured by the plant's root system, accumulates in the deep soil layers. Because some insecticides remaining in the soil have a half-life of about three years, there is an annual accumulation of toxic compounds with negative effects on soil health and beneficial organisms. According to European environmental protection policies (The European Green Deal), it is recommended that by 2030, the quantity of pesticides used for the protection of agricultural crops should be halved. In this case, the permitted quantity of pesticides for use on one hectare will have an approximate value of 50-60 grams.

2. The purpose of the doctoral thesis

The purpose of the addressed topic is to conduct research on reducing the quantity of pesticides used for crop protection by developing new seed treatment processes. To address this challenge*, the research topic aims to contribute with a novel seed treatment approach, allowing protective active substances to become integrated into the internal structure of seeds, providing the embryo with a competitive advantage for internal and external nutrient resources*.

By internally storing active substances, advantages such as reducing environmental contamination with toxic aerosols generated during the mechanical drive of seeds at the time of sowing, decreasing the quantity of toxic residues in the soil, and reducing the half-life of pesticides under the action of solar photocatalysis can be achieved. This approach also resolves issues related to embryo suffocation in soaking treatments and eliminates the loss of minerals through diffusion during seed treatment by immersion.

Storing active substances in the porous matrix of seeds involves replacing intercellular air with gases with liquids carrying the substances of interest through mass transfer based on diffusion in a vacuum. Since mass transfer from the seed to the exterior and vice versa is accompanied by an energy transfer through fragile biological structures, such as cell membranes, *designing the constructive and functional parameters of the vacuum seed treatment equipment represents both theoretical and practical challenges that determine the quality of the treatment.*

The research in the addressed topic is based on the hypothesis that the seed treatment process, including the treatment product within the internal cavities of the seeds, can contribute to reducing the quantity of toxic substances released into the environment compared to the surface treatment method.

3. Proposed objectives

The main objective of the research topic is to identify and describe the physical and biological phenomena that occur during seed treatment using vacuum as a method to accelerate the diffusion of active substances into the porous matrix of seeds. To achieve the main objective of the work, the following specific objectives were necessary:

- study the theoretical elements of the seed treatment process before sowing, aiming for protection against soil and external pests,
- analyze the current state of methods, technologies, and equipment used in the seed treatment process,
- conduct a literature review on the physical properties of seeds and the substances used for their treatment before sowing,
- present a synthetic overview of the factors influencing the seed treatment process, methods, and technologies used in pre-sowing seed treatment,
- present the main theoretical aspects of sorption and desorption processes in the seeds of agricultural crops and the factors influencing them,
- define the working parameters of the seed treatment equipment through physical-mathematical simulations and laboratory experiments,
- physico-mathematical simulation of the seed treatment process by vacuum,
- experimental research on absorption processes in seed treatment by vacuum,
- experimental research on the dose of active substance used in seed treatment,
- experimental research on mass transfer in the seed treatment process,

- numerical and experimental analysis of the seed degassing system in a vacuum,
- identify a set of conclusions leading to the optimization of the cereal seed treatment process by vacuum before sowing,
- disseminate research results on seed treatment by vacuum through the publication of scientific papers in journals and specialized volumes.

4. Activities undertaken

The achievement of the proposed objectives within the research theme relied on conducting theoretical and practical research covering the fields of mechanical engineering and biology. The interdisciplinary nature of the research necessitated the definition of activities covering all stages required for the development of a technology, from the conceptual phase to the validation phase. The research theme begins with defining the specific problems faced by the agricultural sector related to pesticide use and continues with the identification of technological solutions within the mechanical engineering domain, following the series of activities outlined below:

1. Conducting an analysis of the surfaces cultivated with grain cereals in Romania.
2. Estimating the annual pesticide consumption in Romania.
3. Identifying the main causes of soil pollution with pesticides.
4. Describing the methods and techniques for seed treatment.
5. Describing the equipment and apparatus used in seed treatment.
6. Describing the equipment and apparatus used in seed treatment through vacuumization.
7. Describing the basic properties of the solutions and substances used in seed treatment.
8. Describing the physical and technological properties of grain cereal seeds.
9. Identifying and describing the qualitative indicators of seed treatment equipment.
10. Identifying and describing the parameters influencing the seed treatment process.
11. Analyzing sorption and desorption phenomena in the seeds of agricultural crops and the factors influencing them.
12. Mathematical modeling of the seed treatment process.
13. Calculating the material and energy balance in seed treatments.
14. Designing, planning, and implementing seed treatment equipment through vacuumization.
15. Experimentally determining the specific volume of pesticide absorption by seeds treated through vacuumization.
16. Experimentally determining the specific volume of pesticide adsorption by seeds treated through immersion at atmospheric pressure.
17. Numerical and experimental analysis of the seed degassing system in a vacuum.
18. Validating the seed treatment technology through vacuumization using bioassays characterizing seed vigor.

4.1. Analysis of the cultivated areas with grain cereals in Romania

The cultivation of corn (*Zea mays* L.) in Romania covers an approximate land area of 2.5 million hectares, with an average production of 4.8 tons/ha (Table 1). This places Romania below the European average regarding the average production of 7.2 tons/ha. The productivity differences between Romania and European countries are generally caused by variations in precipitation, the spectrum of pests, and the calendar date of sowing. An important factor in mitigating crop losses is seed treatment with plant protection products and nutritional substances that can enhance germination at temperatures below the biological threshold.

Table 1. Dynamics of corn production per hectare and cultivated areas in the years 2010-2019 (MADR according to INS)

Year	Area (thousands of hectares)	Average Production (kg/ha)
2010	2098,4	4309
2011	2589,7	4525
2012	2730,2	2180
2013	2518,3	4488
2014	2512,8	4770
2015	2605,2	3462
2016	2581,0	4159
2017	2402,1	5959
2018	2439,8	7644
2019	2678,5	6502
Average	2515,6	4799,8

Cultivation of wheat (*Triticum aestivum* and *Triticum durum*) occupies an average area of 2 million hectares in Romania's agricultural land, with an average production of 3.8 tons per hectare (Table 2), significantly below the European average of 6 tons per hectare. The main challenges faced by wheat cultivation are caused by low precipitation levels in the Baragan Plain during the sowing period, soil freezing in winter, viral diseases established in autumn, fungal diseases during the intense growth period, and abundant precipitation during harvest. Improving these issues can be achieved through seed treatments with plant protection products and nutrients to increase the seeding depth towards soil layers with higher moisture content.

Table 2. Dynamics of production per hectare and cultivated areas with wheat in the years 2010-2019, according to MADR data after INS

Year	Area (thousands of hectares)	Average Production (kg/ha)
	2162,4	2688
2011	1947,0	3663
2012	1997,6	2652
2013	2104,0	3468
2014	2112,9	3590
2015	2106,6	3780
2016	2137,7	3944
2017	2052,9	4888
2018	2116,2	4793
2019	2168,3	4749
Average	2090,56	3821

Barley (*Hordeum vulgare* L., six-row barley) and hulless barley (*Hordeum distichon* L., two-row barley), according to MADR data after INS (Table 3), occupy approximately 465 thousand hectares of agricultural land in Romania, with an average production of 3.4 tons per hectare. It has become an important crop in recent years due to the development of the fermented beverages sector, where it serves as raw material for enzymes and fermentable biomass. The root system of barley is generally weaker than that of other cereals. The plant is sensitive to nocturnal temperature variations with high amplitude, and below -9°C, the plants may freeze. Considering its biomass accumulation capacity at lower temperatures and shorter biological cycle, the plant can become an option for a second crop within the same growing season. To be used as a second crop, barley requires seed treatments with nutrients and water supply to accelerate germination and, implicitly, ground coverage to prevent evaporation during the warm season.

Table 3. Dynamics of production per hectare and cultivated areas with barley and hulless barley in the years 2010-2019, according to MADR data after INS

Year	Area (thousands of hectares)	Average Production (kg/ha)
2010	515,8	2542
2011	419,5	3170
2012	424,2	2325
2013	495,7	3111
2014	516,0	3319
2015	469,9	3461
2016	481,6	3773
2017	455,0	4186
2018	423,5	4417
2019	448,8	4188
Average	465	3449

Rye (*Secale cereale* L.) occupies an average of 10-12 thousand hectares with a productivity of 2.4 tons per hectare and usually replaces wheat cultivation in areas with harsh pedoclimatic conditions because it has a strong root system that can explore the soil up to depths of two meters. Oats (*Avena sativa* L.) represent an important nutritional source for both humans and animals. In Romania, the cultivated area with oats, according to data recorded by INS for the year 2020, covered 102 thousand hectares with an average production of 1942 kg/ha. Oats cultivated together with vetch produce the spring forage, which is used as green fodder and helps in soil regeneration by fixing atmospheric nitrogen.

4.2. Determination of the annual pesticide consumption in Romania

On a global scale, pesticides are used in extremely varied quantities (fig. 1). For instance, Costa Rica, among the top five exporters of tropical fruits [2], had a consumption of 20.6 kg of pesticides per hectare in 2019 according to statistical data [3], while the United States, the largest exporter of cereals, had an average pesticide consumption of 2.8 kg per hectare in the same year. Romania, based on statistical data (Fig. 2), had a consumption of 0.7 kg of pesticides for the same period. In Romania, the quantity of pesticides (insecticides and fungicides) required for treating cereal seeds is approximately 575 tons of active substance (A.S.), and it will need to be halved by the year 2030.

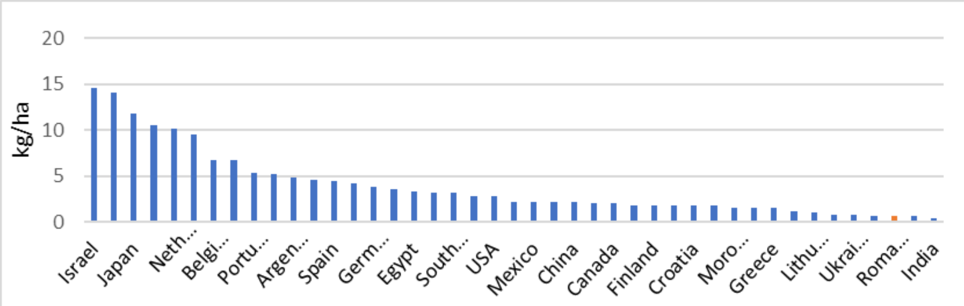


Fig.1. The quantity of pesticides used worldwide in the year 2019

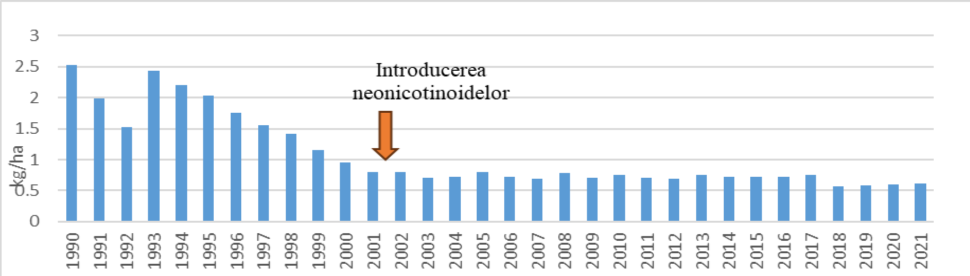


Fig.2. The average quantity of pesticides used per hectars in Romania between 1990-2021 (FAOSTAT)

In the paper "Impact of Seed Treatment with Imidacloprid, Clothianidin, and Thiamethoxam on Soil, Plants, Bees, and Hive Products" [4], the authors analyzed 653 samples over a five-year monitoring period (2018-2022) that could have contained active substances used in seed treatment. The highest number of positive samples was recorded in soil samples (fig.3.), and the presence of substances was even reported in floral organs or pollen. In this case, the biocidal activity of the seed treatment products far exceeded the phenological stage of two to four leaves of plants, when damage from the grey corn weevil (*Tanymecus dilaticollis* L.) can occur. These data demonstrate that at least 20 percent of the treated product mass remained in the soil, and another 20 percent was used ineffectively.

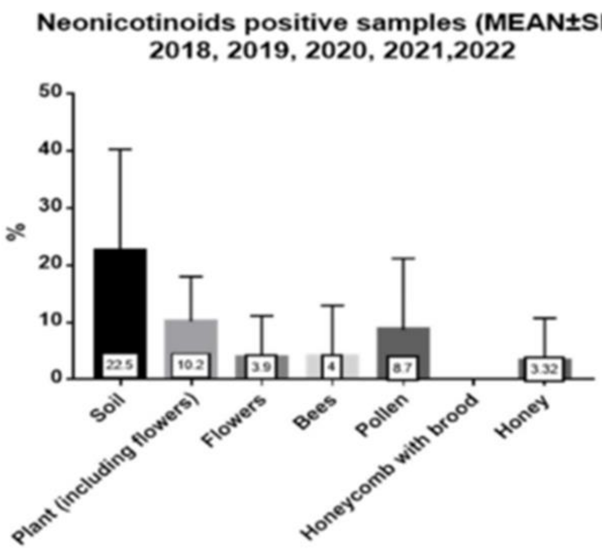


Fig.3. The percentage of positive samples with neonicotinoids

Soil samples were considered positive when the pesticide quantity exceeded the value of 0.01 mg/kg (the quantification limit of the analysis equipment). By performing a calculation based on a sampling depth of 10 cm, there is an accumulation of at least 12 grams of pesticides per hectare in the 0-10 cm layer. Considering that the soil is mechanically processed to a depth of 25 centimeters each year, there would be a theoretical minimum accumulation of 30 grams, which represents approximately 33% of the annual administered insecticide quantity with the seeding of treated seeds. In conclusion, the European Commission's recommendation to halve the amount of pesticides becomes feasible.

4.3. Identification of the main causes of soil pollution with pesticides

The growing need for food and the trend towards reducing labor in plant cultivation have led to the large-scale adoption of chemical methods to control pest populations, regardless of the ecotoxicological implications. Over the past seventy years, it has been observed that pests have developed biochemical and behavioral adaptation to pesticides, resulting in a loss of efficacy of these substances [5], necessitating an increase in the administered dose per agricultural area. Climate change, particularly increased aridity and low precipitation levels, leads to reduced pesticide absorption from the soil with water. The low absorption, coupled with the high half-life of pesticides, promotes the multi-year accumulation of pesticides in the soil. A major cause of pesticide pollution is the cultivation of a single plant species on the same land for several years. Monoculture, especially in the case of cereals, promotes the multiplication of pests and pathogens, resulting in a higher attack and, consequently, the application of a large number of phytosanitary treatments with pesticides.

4.4. Description of methods and techniques for seed treatment

Depending on the desired purpose, seed treatment can be classified into four types: (1) treatments to break dormancy; (2) treatments to promote growth and development; (3) curative treatments against pests and pathogens; and (4) preventive treatments ensuring seed and plant protection against diseases and pest attacks.

Treatments to break dormancy involve soaking seeds in water to initiate biochemical reactions by activating endogenous enzymes. Treatments for growth and development involve adding a mass of nutrients or growth hormones to the seed mass. Curative treatments carried out on seeds generally aim to process them for the food industry or long-term storage. This type of treatment uses substances that can be removed from the seed mass, such as aluminum phosphide or ethyl formate in combination with carbon dioxide [6]. Preventive treatments on seeds are performed to protect them in the early vegetative stages in case of potential attacks by harmful agents.

Based on the aggregate state of the treating agent, methods are distinguished as follows: (1) gas treatments (fumigation); (2) dry treatments (with powders); (3) liquid treatments; and (4) mixed treatments with liquids and powders.

Seed fumigation involves ventilating a stream of air containing insecticides in vapor form over a seed mass. This type of seed treatment is mainly used in enclosed silos. In recent years, this type of treatment has gained an ecological trend as studies with promising results have emerged regarding insect control in seed and fruit/vegetable storages using volatile substances with low toxicity.

Dry treatment is applied to a large number of crops, including cereals, vegetables, and technical crops. It involves applying active substances in solid form conditioned as powders to the surface of the seeds. Additives such as activated charcoal or polyethylene glycol can be used along with the active substances. The method of treating seeds with dry powders has been increasingly used in recent years due to the introduction of non-toxic treating agents (micronutrients, growth promoters, natural enzymes, nitrogen-fixing microorganisms).

Wet treatment is a method that involves soaking the seeds in the treating solution or suspension. Usually, for this type of treatment, a volume of 100 liters of water per ton of seeds is required. In terms of effectiveness, wet treatments also have an immediate curative effect, destroying microbial forms of resistance that have entered the seed cavities. The coverage degree is higher than in the case of dry treatments. The method becomes challenging when seed dehydration is necessary because it involves capturing and recycling eliminated pesticides along with water vapor.

Semi-liquid treatment (Slurries) or slurry treatment is based on mixing a small amount of water with the treating product, forming a dense suspension. This is introduced into a mixing machine so that it can be dosed well. Machines designed for this type of treatment have a seed flow control device, a reservoir with an agitator to keep the mixture sufficiently homogeneous, a dosing device, and mixing equipment. Through this treatment, seeds are covered with a fairly dense layer of suspension that not only disinfects the seed surface but also protects the plant against soil pathogens for a longer period. The small amount of water, generally 3-8 liters per ton of seeds, eliminates the need for seed drying. This seed treatment method is widely used in industrial installations for treating corn. A newer method adapting to seed slurry treatment is glazing and pelleting seeds. In this method, liquids with adhesive properties, filling powders, and active ingredients are used.

The main techniques for seed treatment involve the use of substances, electromagnetic waves, electric currents, magnetic fields, and pressure variations through acoustic pulsations. These techniques are based on the dose of the treating agent, the exposure time of the seed, and the induced effect. For the induced effect on seeds to be beneficial, the existence of synergy or an optimal ratio between the dose of the treating agent and the exposure time is necessary.

Hot water treatment of seeds and calluses is known as a method for inactivating viruses transmitted through seeds. In the case of carrot, cabbage, celery, parsnip, and lettuce seeds, hot water treatment has been very successful in controlling pathogens such as *Alternaria spp.*, *Phoma spp.*, *Peronospora spp.*, *Septoria spp.*, and *Xantomonas* at a temperature of 50°C for 30 minutes [7]. Table 4 presents the temperature values and exposure times for hot water treatments.

Table 6. Parameters for hot water seed treatment, [4]

Vegetables	Temperature of the water treatment bath (ºC) and duration (min.)			
	47,7 (ºC)	48,8 (ºC)	50 (ºC)	51,6 (ºC)
Broccoli			20	
Brussels sprouts			25	
Cabbage			25	
Chinese cabbage			20	
CarrotMorcov			15 - 20	
Cauli flower			20	
Celerz	30			
Collard greens			20	
Cucumbers			20	
Eggplant			25	
Garlic		20		
Turnips			20	
Rutabaga			20	

Vegetables	Temperature of the water treatment bath (ºC) and duration (min.)			
	47,7 (ºC)	48,8 (ºC)	50 (ºC)	51,6 (ºC)
Lettuce	30			
Mustadt greens			15	
Bell peppers				30
Radishes			15	
Swedish turnip			20	
Spinach			25	
Tomatoes			25	

Ultraviolet (UV-C, 100–280 nm) radiation treatment has yielded significant results in the germination and vigor of soybeans, wheat, and pine using a dose of 43.20 kJ/m^2/day and sunflowers using a dose of 2.88 kJ/m^2/day [9]. The beneficial effect of UV radiation on pine was somewhat expected, as the plant belongs to the Gymnosperms group, whose ecosystem is located at high altitudes where UV radiation is higher.

Plasma treatments represent a relatively new field, as they require complex working equipment, involving control of vacuum, exposure time, potential energy difference between electrodes (1-10 eV), electromagnetic radiation ranging from 150 nm to 1100 nm. Identifying measurable biological parameters is challenging, except for germination and root growth bioassays. By using non-thermal plasma, Zhang et al. [10] achieved visible results regarding the size of pepper seeds. The use of testing equipment and cold plasma seed treatment technology originates from the development of plant growth programs in extreme conditions within space programs.

4.5. Description of the equipment and apparatus used in seed treatment

The addition of a quantity of product intended for seed treatment onto their surface or inside them involves a series of working processes such as weighing the seeds, dosing the product equivalent to the seed mass, homogenizing them, and conditioning the treated seeds. The complexity of the seed treatment process is determined by the type of equipment used and the legislative requirements in the field. The simplest seed treatment process is based on equipment (Fig. 4) consisting of a supporting metal frame, a cylindrical mixing drum mounted eccentrically on a horizontal axis, manually operated through a handle.

Fig. 4. Manually operated seed treatment apparatus, [11]

The working processes become particularly complex when obtaining treated seeds must meet stringent standards regarding seed quality, worker protection, environmental protection, and the management of substance residues. Such equipment (fig.5.) is composed of electronically controlled seed and seed treatment product feeding elements, the homogenization drum equipped with accessories for internal washing and cleaning, the weighing and packaging compartment, and a system for capturing and storing residues produced during operation.

Fig.5. Industrial seed treatment equipment, On Demand, Bayer, [12]

A. Rotary Drum Seed Treatment Equipment

Belonging to the category of rotary drum seed treatment equipment are the Romanian MTS seed treatment equipment (fig.6.) and the Panogen equipment optimized for labor protection. The MTS1 seed treatment equipment is of Romanian production and is capable of treating seeds simultaneously or separately with powders and liquids.

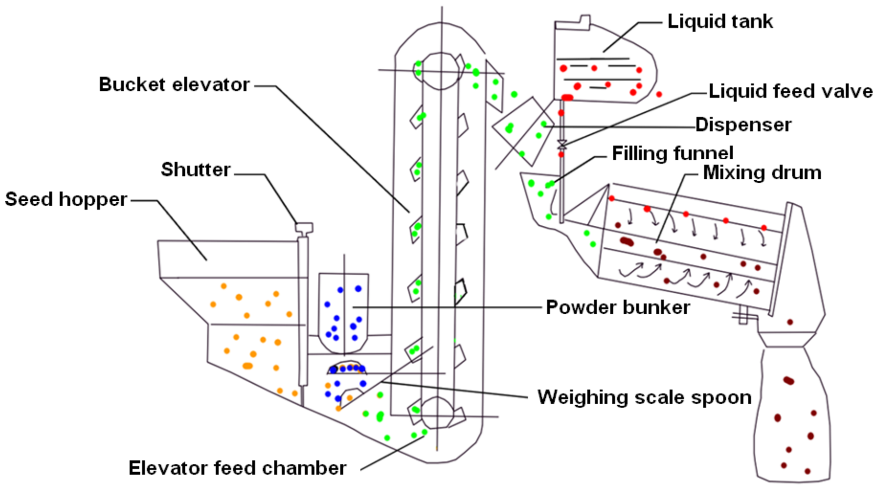


Fig.6. Seed treatment machine (STM), [13]

The equipment is composed of the seed hopper, gate, the first powder dosing scale, powder hopper, bucket elevator, the second scale for liquid treatment dosing, treatment liquid tank, and mixing drum. Seeds from the equipment's hopper are controlled released through a gate that regulates the opening surface of the discharge hole into a weighing and dosing spoon. When the weighing spoon is filled, it performs a downward rocking motion, opening the hole at the base of the powder hopper and releasing a quantity of powder, followed by emptying and lifting back to the initial position. This oscillating action controls the seed flow and the amount of powder product. After weighing, the seeds fall into a bowl where they are taken up by a bucket elevator and poured into a vessel attached to a swinging arm with a counterweight. When the vessel is filled, it performs an emptying motion and drives a dosing tap for the treatment liquid located at the base of the liquid vessel. The mixture of seeds, powder, and liquid is directed through a chute into the rotating drum, which performs the homogenization and discharge operation into a bag. The mixing time is controlled by the rotation speed of the drum, its inclination, and the force of the spring pressing on the discharge flap.

The Panogen equipment (Panogen – methylmercury dicyandiamide – $C3H6HgN4$, fig.7.) was intended for treating seeds with mercurial products. Because these products are highly toxic to operators, the equipment is equipped with an exhaust fan that creates an air pressure inside the mixing drum lower than atmospheric pressure. Although mercury-based compounds were banned in 1982, the equipment still has practical interest. Constructively, the equipment consists of a seed hopper, seed weighing scale controlling the liquid product flow, liquid vessel filling pump, mixing drum, and exhaust fan for toxic vapors and particles. The seed treatment principle is similar to rotary drum equipment, with the difference being the extraction and removal of toxic particles and gases from the mixing drum.

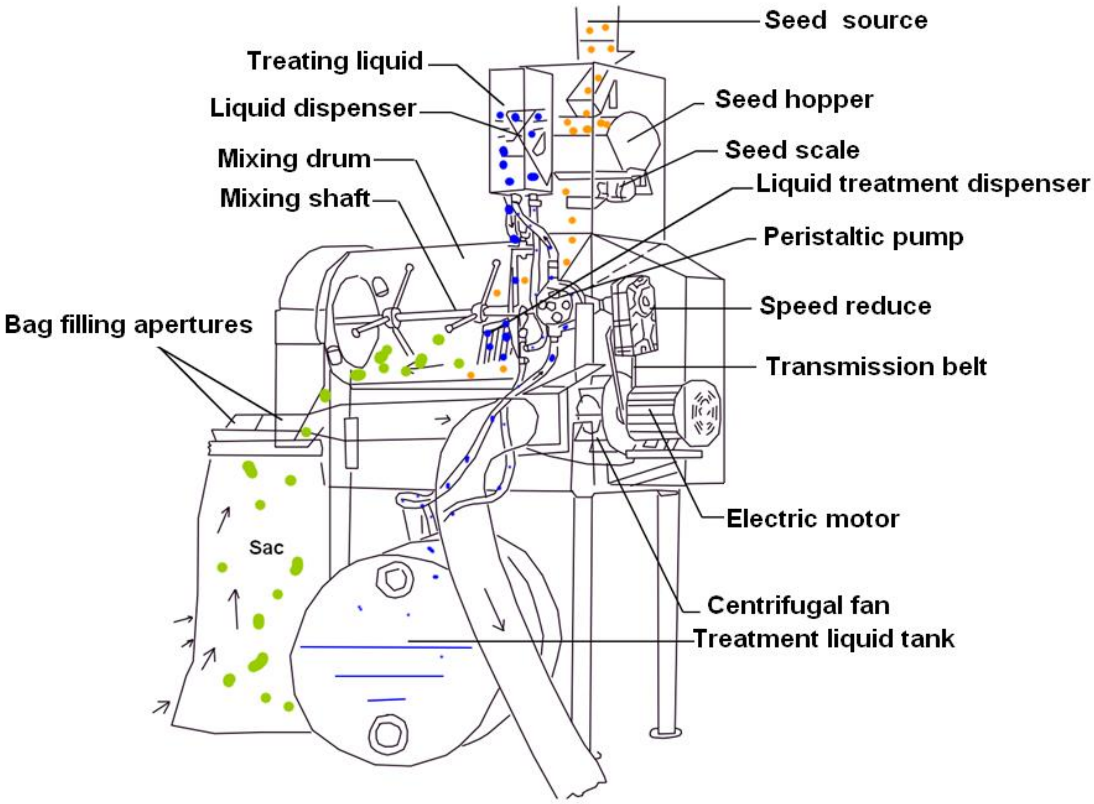


Fig.7. Schematic diagram of the optimized liquid seed treatment machine operation

Also belonging to the category of rotary drum seed treatment equipment is the equipment designed by John J. Simmons (fig.8.), patented in 1984. Unlike the MTS and Panogen equipment, this equipment has two rotary drums: one dedicated to mixing seeds with liquid or adhesive liquids, and the other for mixing seeds moistened with a powdered product. Seed feeding and transport occur in a cascade from the first drum to the second drum. The seed and powder flow rate are controlled using a star-shaped rotor driven by a variable-speed electric motor, while the liquid flow rate is controlled by a solenoid valve.

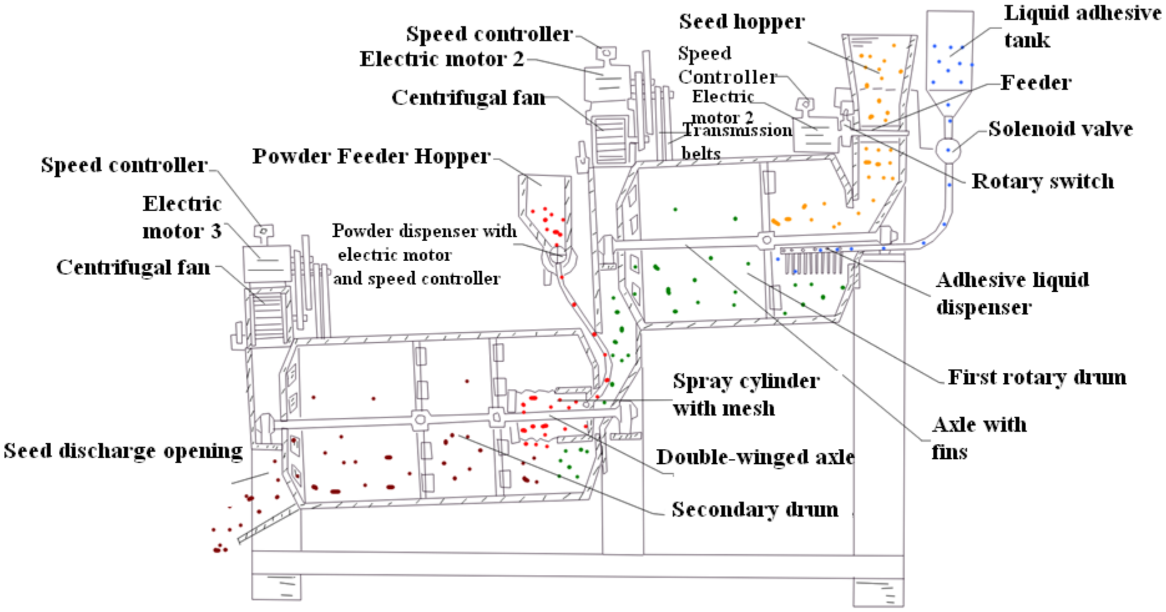


Fig.8. Schematic diagram of the two-stage operation of the seed treatment machine with powders and liquids, according to U.S. Patent No. 4,465,017 from 1984 by John J. Simmons, [14]

B. Seed Treatment Equipment with Centrifugal Discs

The seed treatment equipment patented by Lund in 1999 [15] (fig.9) introduces a new element regarding the flow of seeds and treatment liquids inside the installation. The seed dressing apparatus consists of an internally open enclosure equipped with loading and unloading openings, a dual shaft (concentric) driven by two motors with different rotational speeds that set-in motion a seed distributor and a disc that produces an air curtain coupled with a liquid dispersal head. The seed dosing arm is located on the outer axis and consists of a truncated cone with multiple radially arranged blades forming trays that narrow towards the edges. Under the conical seed distributor, there is a rotating disc on the inner axis, which, at a rotation speed of 1000 rpm, produces an air flow similar to a curtain that uniformly disperses the seeds to be sprayed on all sides with treatment liquids. Also on the inner axis is the liquid dispersal head, consisting of a rotating conical vessel with perforations on the outer walls and a fixed cap through which the liquid supply pipes are connected. Under the action of centrifugal force on the surface of the dispersal head, fine accelerated droplets are released, hitting the seeds. The volume of the dispersed liquid and the size of the droplets can be controlled by introducing an interchangeable deflector screen inside the dispersal head through which slots of different widths are dispensed. The seeds are dosed with the help of a conical shaft equipped with fins that delimit a known volume. By rotating the conical shaft with fins, equal quantities are extracted from the seed hopper. Depending on the speed of the electric motor that drives the distribution cone, the working flow rate of the treatment installation can be adjusted. The rotation speed of the conical shaft for dosing the seed flow is synchronized with the control valves of the liquid flow. A novelty in this type of installation is the formation of aerosols by centrifugal forces induced by the liquids through the head of the inner axis, which has small-diameter nozzles. The higher the rotation speed of the inner axis, the finer the mist of aerosols. The secondary axis is equipped with a rotating disc that forms an air curtain directing the seeds uniformly into the mixing chamber. In this installation, the seeds are not subjected to friction as in drum systems.

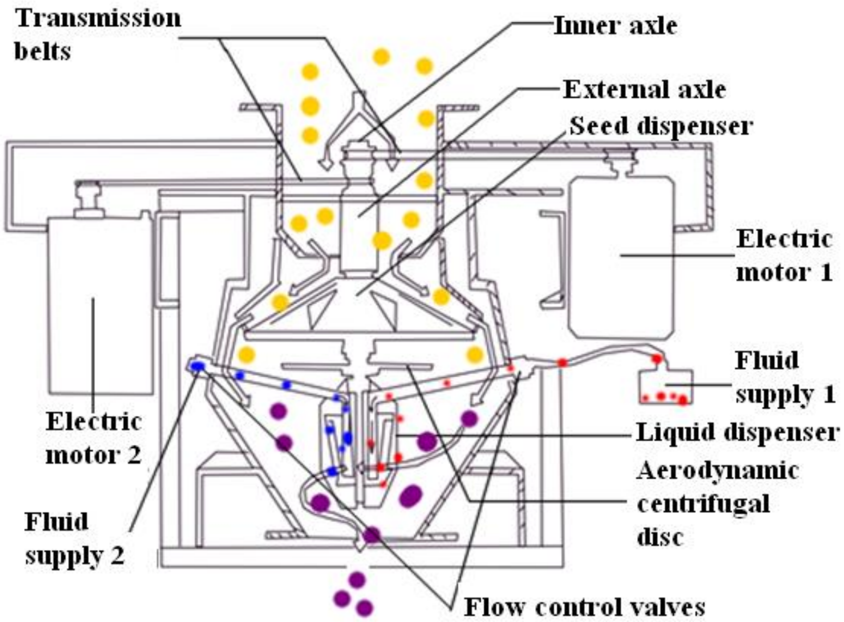


Fig.9. Schematic diagram of the optimized liquid seed treatment machine, designed to reduce seed degradation through abrasion

Equipment such as Wallez and MTS-4 (fig.10) also falls into the category of seed treatment equipment with centrifugal discs. The distinctive element of this equipment (MTS-4) is the presence of a bucket elevator and the homogenization of the product with the seeds in two stages through the use of two centrifugal discs.

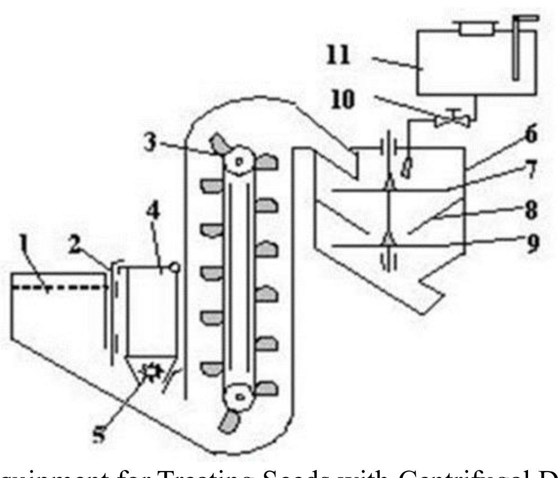


Fig.10. Equipment for Treating Seeds with Centrifugal Discs, [16]
1. seed hopper, 2. sliding valve for adjusting seed flow, 3. bucket elevator, 4. powder vessel, 5. star dosing device, 6. homogenization chamber, 7. first centrifugal disc, 8. funnel, 9. second centrifugal disc, 10. control valve for treatment liquid flow, 11. vessel with treatment liquid.

C. Other Seed Treatment Equipment

In Figure 11, the operating diagram of a high-flow seed treatment equipment is presented, which can be mounted above transport containers. The liquid treatment aerosols are formed hydraulically using high-pressure pumps and atomization nozzles. The installation is equipped with pneumatic control of intake and exhaust ports. The synchronization of the seed quantity with the treatment liquid is achieved using a grain flow measuring device coupled with control valves for the sprayed fluid and through the composition of the treatment liquid. Inside the installation, the seeds are agitated and twisted on all sides by the force of the liquid jet. The seeds capture a small amount of liquid on the surface, and the rest is recovered by a pump that draws the accumulated liquid in a specially designed area. Through this treatment method, the treated seeds do not require subsequent drying treatment.

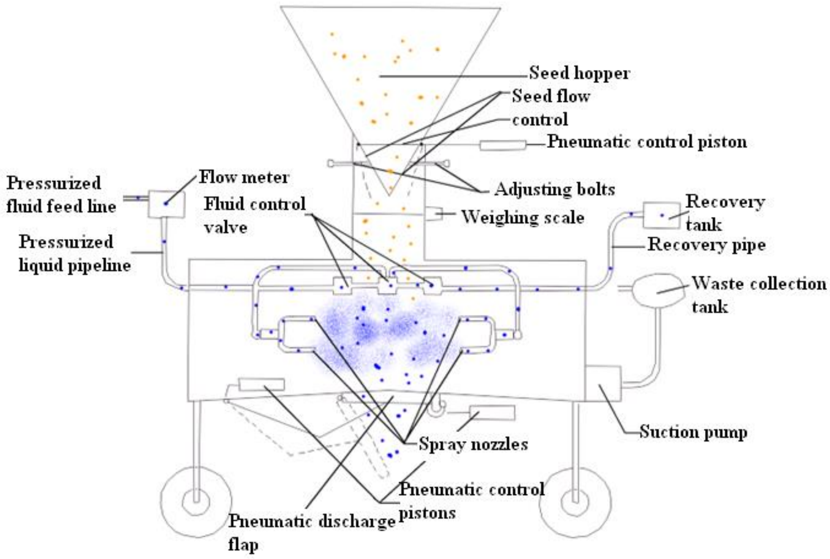


Fig.11. Schematic diagram of the optimized liquid seed treatment machine for high seed throughput, [17]

Treating seeds through exposure to non-ionizing electromagnetic radiation or high-density magnetic fields is still an experimental method. Such equipment is presented in Figures 12 and 13.

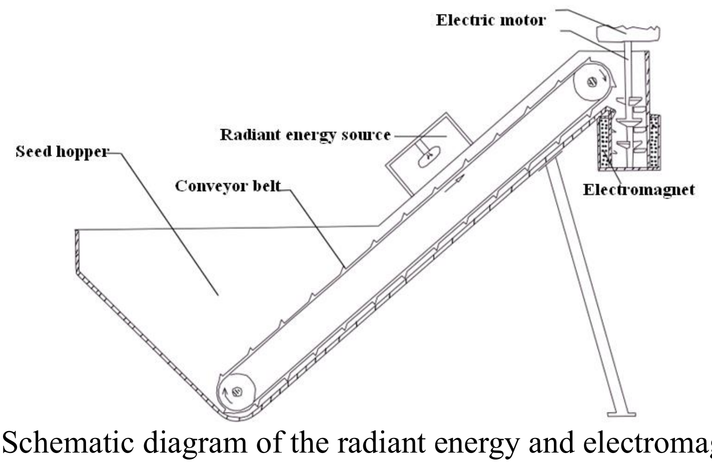


Fig.12. Schematic diagram of the radiant energy and electromagnetic field seed treatment machine operation, [18]

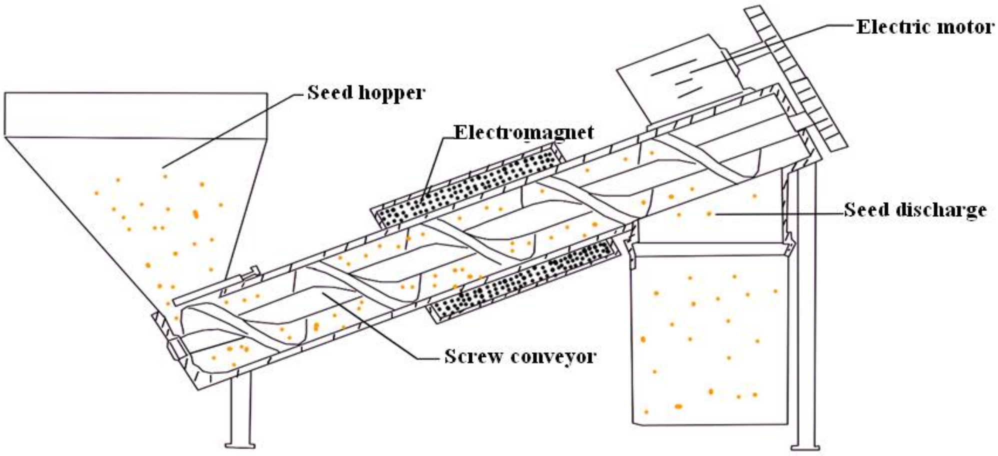


Fig.13. Schematic diagram of the operation of the seed treatment machine with an electromagnetic field, [19]

An equipment for seed treatment by immersion that can shorten the execution time is presented in Figure 14, where a shock wave emitter (acoustic or ultrasonic) is used to induce oscillating cavitation in seeds, resulting in accelerated absorption of the treating liquid. The patent holder claims positive results using only water as the treating liquid. The dosing of frequency and amplitude of pressure pulsations in this equipment must be well controlled, as this process is typically used in the disintegration of cell membranes.

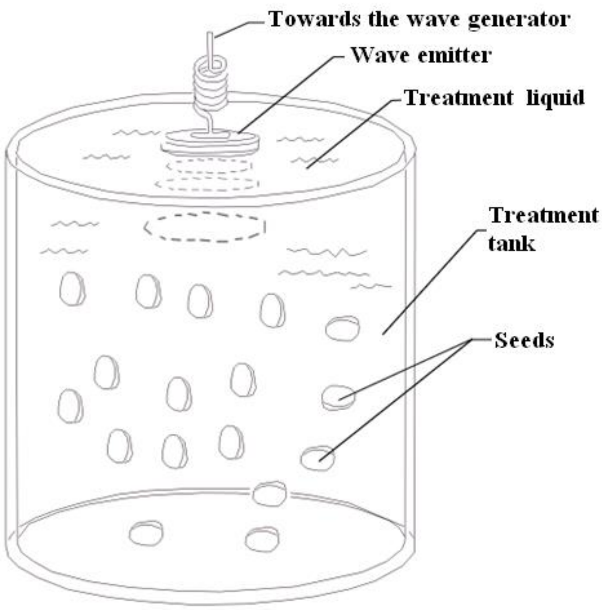


Fig.14. Schematic diagram of the operation of the seed treatment machine through shockwave-assisted immersion, [20]

The technology of treating seeds using cold plasma began to be known in 2015 with the publication of the scientific paper "Cold Plasma: A novel Non-Thermal Technology for Food Processing" [21], even though the equipment was developed in 2005 by Changzhou Zhongke Changtai Plasma Technology Co., Ltd, under the name "HD-2N Cold Plasma Seed Processor" (fig.15).

Fig.15. HD-2N Cold Plasma Seed Processor (Changzhou Zhongke Changtai Plasma Technology Co., Ltd)

According to the manufacturer, the characteristics of the equipment are as follows: installed power 500 W, discharge current frequency 13.56 MHz, gas pressure in the chamber 80-150 Pa, and productivity 80-120 kg/h. The operating principle of the equipment involves the production of reactive oxygen species through the dissociation of water. These molecules have antiseptic properties and can destroy microorganisms present in the seed mass, as well as neutralize volatile toxic substances [21].

4.6. The description of the equipment and apparatus used in seed treatment through vacuumization

The equipment for treating seeds using acoustic and ultrasonic pulsations presented in Fig. 14 and the seed treatment equipment with cold plasma presented in Fig. 15 use vacuum in different ways. In the working process of the first equipment, the vacuum appears for very short periods due to the cavitation phenomenon generated by acoustic or ultrasonic pulsations. The presence of the vacuum is not continuous but pulsatory, and the frequency of vacuum occurrence is linked to the frequency of oscillations of the wave emitter. The volume of the cavity where the vacuum occurs is determined by the amplitude of the emitted wave. When the vacuum bubbles collapse, shockwaves are formed, which have a destructive effect on the materials they come into contact with. The repetitive effect of shockwaves promotes the mechanical destruction of microbes that can infect the surface or interior of the seeds. The choice of frequency and amplitude of shockwaves is critical because during the process, embryo cells in the seeds can also be destroyed.

In the working process of the second equipment (Fig. 15), the presence of vacuum is a mandatory condition for the appearance of cold plasma between the anode and cathode of the plasma generator. Since the kinetic energy of accelerated particles can reach up to 10eV, ionizing electromagnetic waves appear, inducing the formation of reactive oxygen species. Two of these species are hydrogen peroxide and ozone. It is recognized that hydrogen peroxide and ozone have antimicrobial effects and are commonly used in medical practices to disinfect surfaces.

As part of the doctoral thesis, equipment for seed treatment using vacuum as a method to facilitate the transport of liquid substances in the porous matrix of seeds was proposed. The schematic principle of this equipment is presented in Fig. 16.

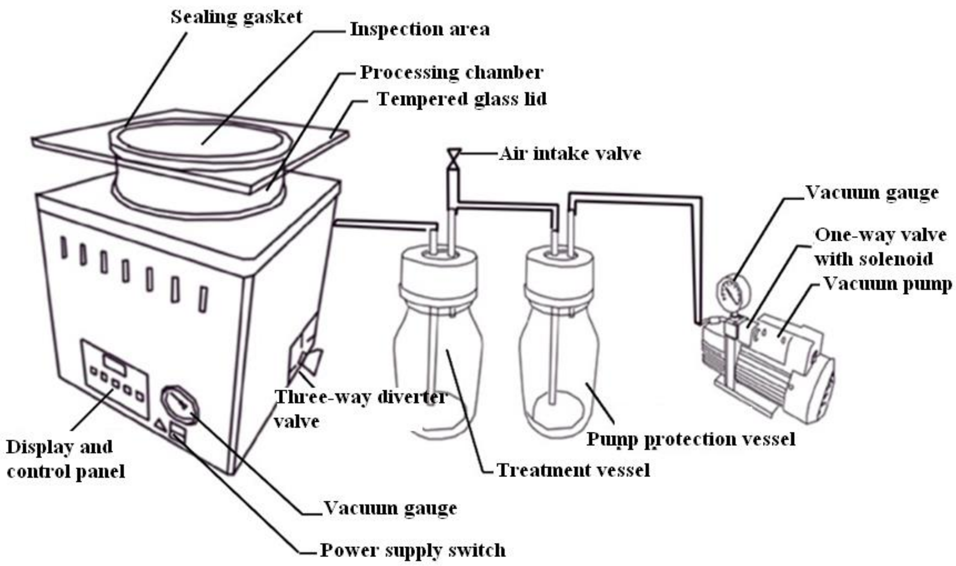


Fig.16. Seed treatment equipment using vacuum

The equipment consists of four main components: the treatment chamber, the liquid treatment reservoir, the vacuum pump, and elements for process protection and control. The working process of the equipment involves two distinct actions. In the first action, air from the internal pores of the seeds is evacuated through a vacuum, and in the second action, there is accelerated absorption of the treatment liquid in the place of the evacuated air. The treatment process has a short duration, up to 10 minutes per seed batch.

4.7. Description of the basic properties of solutions and substances used in seed treatment

Seed coating involves the application of a fine powder or a liquid containing dissolved or suspended solids on the surface of seeds, forming a continuous layer with uniform thickness. In the specialized literature, seed coating can also be referred to as pelleting, drageeing, granulation, and filming. Pelleting involves applying a quantity of solid material to the seed's surface with the aim of increasing volume or weight for precise sowing. Drageeing seeds is similar to pelleting and is especially applied to small-sized seeds. Seed granulation is perhaps one of the oldest methods of seed treatment, involving embedding seeds in a thick mass that, after drying, takes on the appearance of balls. Coating seeds through filming involves covering the seed with a very thin layer. Generally, the deposited film serves an adhesive or coloring purpose and can constitute either an independent treatment or an intermediate step in a complex coating process. The technology of seed treatment through coating or dressing involves the use of liquids conditioned to be compatible simultaneously with mechanical equipment, seeds, and the final purpose of the treatment. The main identified technological properties of fluids used in seed coating treatments are:

- Density of seed treatment fluids;
- Viscosity of liquids;
- Adhesiveness;
- Corrosiveness;
- Color;
- Odor;
- pH of liquids.

4.8. Description of the physical and technological properties of cereal grains

Currently, the seed coating with treatment products falls within the range of 300 g/ton to 6 kg/ton. The maximum loading capacity of seeds with plant protection products depends on seed properties such as size, mass, density, surface roughness, porosity, moisture, and imbibition. In some cases, utilizing the maximum loading capacity of seeds with plant protection products may negatively impact seed physiological processes such as respiration, dormancy, and germination. Table 7 presents the main physical and technological properties of seeds from various plant species cultivated for human and animal consumption.

Table7. Main physical and technological properties of seeds, [22]

Cereale	Mass of 1000 grains (g)	Water content (moisture) (%)	Apparent density (kg/m^3)	Porosity (%)	Length (mm)	Width (mm)	Thickness (mm)	Spherical index
Barley	30-50	9,7-10,7	618	39,5-57,6	8,4	3,6	2,8	0,52
Rape	5-6	6,5-6,7	669	38,4-38,9	1,8	1,7	1,7	0,96
Maiye	250-300	9-15	721	40,0-44,0	9,4	8,2	5,1	0,78
Linen	4-7	5,8	721	34,6				
Oat	30 - 45	9,4-10,3	412	47,6-55,5	11,5	3,1	2,6	0,39
Rice	15-20	11,9-12,4	579	46,6-50,4	7,3	2,3	2,2	0,46
Rye	25-35	9,7	721	41,2	7,2	2,9	2,6	0,53
Soy	140-150	6,9-7	722	33,8-36,1	8,2	6,6	5,6	0,82
Wheat	41 - 45	9,9	722	39,6-42,6	6,7	3,2	2,9	0,59

4.9. Identification and description of qualitative indices of seed treatment equipment

Highlighting the differences between seed treatment equipment is a fundamental condition in choosing the type of equipment required for a seed treatment company. Therefore, besides the economic criterion in selecting a type of equipment, understanding the equipment's performance is necessary. Within the thesis, the main qualitative indices of seed treatment equipment, including the equipment treating through vacuum usage, were identified. These indices (Table 8) have been classified into four categories of qualitative indices as follows: (1) qualitative indices related to the construction of the equipment; (2) qualitative indices related to the treatment process; (3) qualitative indices related to the quality of the finished product; and (4) indices related to the quality of the treatment.

Table 8. Qualitative indices of seed treatment equipment

Constructive Quality Indices	**Process Treatment Quality Indices**	**Finished Product Quality Indices (Treated Seeds)**	**Treatment Quality Indices**
Size	Quantity of product added to seed mass	Physical-mechanical integrity of seeds	Germination percentage
Installed power	Uniformity of the product on the surface or inside the seeds	Product content found in seed mass	Growth speed

Constructive Quality Indices	**Process Treatment Quality Indices**	**Finished Product Quality Indices (Treated Seeds)**	**Treatment Quality Indices**
Treatment flow rate	Product losses due to adhesion to the working surface	Water content	Sprouting percentage
Energy efficiency	Degree of recovery of unused products	Dust content in seed mass	Treatment effectiveness
Chemical and biological safety level	Operational difficulty level of the equipment	Storage time	Duration of beneficial treatment effects
Vacuuming speed	Process duration		
Percentage of achieved vacuum	Specific energy consumption per kilogram of seeds		

4.10. Identification and description of parameters influencing the seed treatment process

In the transformation process of seeds from freshly harvested products to an industrial product with improved agricultural properties, several factors are involved, including the seeds, the treatment product, the equipment performing the treatment, existing environmental and bio-security conditions, and the treatment protocol. To create an overview of the main factors involved in the seed treatment process, parameters were categorized into five types of factors (Table 9):

1. Biological factors related to the morphological and physiological properties of the seeds;
2. Physico-chemical factors characterizing the product used to treat the seeds;
3. Atmospheric conditions in which the treatment is carried out;
4. Technical specifications of the seed treatment equipment;
5. Protocol for the seed treatment process.

Table 9. The main parameters influencing the seed treatment process

Biological factors (seeds)	Physical-chemical factors (treatment product)	Atmospheric factors	Constructive factors	Process factors (working parameters)
Seed geometry	Quantity of product to be treated	Atmospheric humidity	Constructive dimensions of equipment	Technological volume
Seed mass	Water content of the product to be treated	Atmospheric pressure	Equipment power	Percentage load of equipment
Seed density	Concentration of active substance in the product to be treated	Temperature	Productivity (flow rate of treated seeds)	Homogenization time
Seed porosity	Product viscosity	Water vapour pressure deficit	Resistance of biosecurity filters	Homogenization rate
Soaking ability	Latent heat of vaporization	Oxygen concentration	Mass of unused product	Air pressure in the

Biological factors (seeds)	Physical-chemical factors (treatment product)	Atmospheric factors	Constructive factors	Process factors (working parameters)
				homogenization chamber
Tolerable minimum and maximum temperatures	Odor		Energy efficiency of equipment	Working temperature
Specific heat of seeds	Color			

4.11. Analysis of sorption and desorption phenomena in agricultural crop seeds and the influencing factors

The phenomenon of water sorption or the treatment liquids absorption by seeds, known agronomically as seed imbibition, is an inverse process to seed dehydration and can occur over varying periods, ranging from a few hours to several days. The rate of water sorption within the seed under normal atmospheric conditions is dependent on the water's state of aggregation present around the seed. If the water is in liquid form, the seed imbibes through a process called capillary penetration limited by alveolar air evacuation. Because the seed is enveloped in an impermeable membrane, water sorption in the seed competes with the expulsion of air from the alveolar spaces. The imbibition rate in this case depends on the difference in affinity of the molecules in the biological structure for water and air. An indicator of this affinity can be the water activity in the substrate:

$$a_w = \frac{p}{p*} \tag{5.3}$$

Where: - aw represents water activity, p - is the vapor pressure at equilibrium, p∗ - is the partial pressure of pure water vapor at the same temperature.

In the presence of water around the seed in the form of vapor, seed imbibition occurs over long periods, on the order of days. The mass transfer of water vapor takes place through temperature and humidity fluctuations by capturing vapors at the dew point. This phenomenon is demonstrable through the analysis of long periods from sowing to germination in decades with low precipitation. The dew point temperature can be calculated using the following relationship:

$$T_{dp} = \frac{\frac{c \ln(P_a(T))}{a}}{b - \frac{\ln(P_a(T))}{a}} \tag{5.4}$$

where: T_{dp} - dew point temperature (°C); C - 257,14 (°C); b - 18,678; a – 6,1121 (mbar); P_a - actual water vapor pressure (mbar); T – temperature (°C).

The desorption of water from seeds is achieved through energy consumption by transpiration and evaporation. To transition from a liquid to a gaseous state, an amount of energy equal to the latent heat of vaporization of water is required. The presence of water in seeds triggers the biochemical combustion of starch, generating heat that drives the transpiration of water from the seeds into the atmosphere. The evaporation of water from seeds occurs when the atmosphere receives a quantity of solar energy, increasing its vapor saturation capacity and creating a vapor deficit in the seed substrate. The most commonly used equation to approximate the phenomenon of evapotranspiration over a 24-hour period is the FAO Penman-Monteith equation [24]

$$ET_0 = \frac{\Delta(R_n - G) + \rho_a \cdot c_p(\delta_e)\, g_a}{\left(\Delta + \gamma\left(1 + \frac{g_a}{g_s}\right)\right) L_v} \tag{5.1}$$

where: ET_o – the volume of water lost through evapotranspiration (mm/s); Δ – the slope of the vapor pressure curve (kPa/°C); R_n – net radiation, the flux of external energy source (W/m^2); G – soil energy flux (W/m^2); ρ_a – density of dry air (kg/m^3); c_p – specific heat of air (J/kgK); δ_e – vapor pressure deficit (Pa); g_a – air conductivity or atmospheric conductance (m/s); γ – psychrometric constant ($\gamma \approx 66$ Pa/K); L_v – latent heat of evaporation ($L_v = 2453$ MJ/m^3; L_v = 681.388,9 W/m^3); g_s – stomatal or grain conductance (m/s).

The mathematical foundations regarding the water sorption process in plant seeds were established starting in 1943 and have continued to the present day. Determining the amount of water absorbed by seeds (Ue) is an essential requirement in the agri-food industry and biological studies related to seed germination. Most mathematical models for determining the amount of water absorbed by seeds are based on the temperature at which the process occurs and the water activity in the substrate (aw). In the specialized literature, approximately 10 mathematical models have been identified that can determine the maximum amount of water absorbed by seeds. The determination coefficients R^2 of the mathematical models have ranged from a maximum of 0.996 to a minimum of 0.966 between experimental and calculated values. This demonstrates that the subject of water absorption has been studied for a long time (72 years), and theoretical results do not differ significantly from experimental ones. In the baking and fermentation industries, the correct dosing of water in compositions (dough and must) represents determining factors for the quality of products and the shelf stability period.

1. Harkins-Jura 1943 [48] $R^2 = 0,990$

$$U_e = \frac{\exp(2,965 - 0,0059\,T)}{0,620 - \ln(a_w)} \tag{5.6}$$

2. Oswin Model [89], $R^2 = 0,987$

$$U_e = \frac{15,062 \pm 0,143T}{\frac{(1-a_w)^{\frac{1}{3,014}}}{a_w}} \tag{5.7}$$

3. GAB Model [8], $R^2 = 0,979$

$$U_e = \frac{5,59\left(\frac{465,31}{T}\right)a_w}{[1 - 0,835a_w]\left[1 - 0,835a_w + \left(\frac{465,31}{T}\right)a_w\right]} \tag{5.8}$$

4. Henderson-Thompson, [115], $R^2 = 0,976$

$$U_e = \left[\left[\frac{\ln(1-a_w)}{[-0,00027(T+56,361)]}\right]\right]^{\frac{1}{1,232}} \tag{5.9}$$

5. Modified Halsey [55], $R^2 = 0,989$

$$U_e = \left[\frac{\exp(5,608 - 0,0317T)}{-\ln a_w}\right]^{\frac{1}{2,151}} \tag{5.10}$$

6. Chung Pfost [92], $R^2 = 0,996$

$$U_e = 37,41 - 6,23\ln[-(T + 49,64)\ln(a_w)] \tag{5.11}$$

7. Copace [34], $R^2 = 0,966$

$$U_e = \exp[1,267 - (0,0077T) + (2,514\exp(a_w))] \tag{5.12}$$

8. Henderson [99], $R^2 = 0,970$

$$U_e = \left[\frac{\ln(1-a_w)}{[-0{,}000075(T+273{,}15)]}\right]^{\frac{1}{1{,}4195}} \tag{5.13}$$

9. Smith [33], $R^2 = 0{,}986$

$$U_e = 8{,}9201 - (0{,}1481T) - 9{,}010\ln(1-a_w) \tag{5.14}$$

10. Sigma-Copace [116], $R^2 = 0{,}966$

$$U_e = \exp[0{,}3802 - (0{,}0080T) + (1{,}2833a_w)] \tag{5.15}$$

4.12. Mathematical modeling of the seed treatment process

Calculation of the power required for the rotary drum seed treatment process

In their work, Liu et al. (2016) [24], propose a mathematical model for calculating the power absorbed by a rotating drum oven that can be likened to the equipment used for treating seeds in a rotary drum. The discrepancy between the calculated value and the experimentally obtained one was 3%. The proposed relationship is as follows:

$$N = 0.383 \cdot 10^{-6} L D^{0.351} \theta^{1.293} (n\,\rho)^{0.117} \left(\frac{m}{\beta}\right)^{0.883} \tag{4.1}$$

where: N – absorbed power (kW), L – drum length (m), D – internal drum diameter (m), θ – angle of response (relative rest) of the material, n – drum rotation speed (rpm), ρ – apparent density of the material (kg/m^3), m – feed rate (kg/h), β – longitudinal inclination angle of the drum relative to the horizontal.

The mathematical determination of seed movement is important as it can provide information about the mechanical stress to which the seeds are subjected. Based on the individual movement of the seeds, the power absorbed by the seed treatment equipment can be calculated using the discrete element method.

In the paper titled "Investigating DEM of Absorbed Power for Material Movement in Rotary Drums with Axial Offset," the authors propose a model for determining the total power absorbed by the equipment during the process by summing the power absorbed by each particle in the material mass that undergoes lifting, rolling, and falling movements [25]. The parameters of the DEM simulation performed by the mentioned authors include drum diameter, drum length, rotation speed, inclination angle of the drum's central axis relative to the horizontal, filling percentage, particle density, particle diameter, stiffness modulus G, Poisson's coefficient, internal friction coefficient, external friction coefficient, and critical damping coefficient. Using the series of relationships (4.5), the calculation of the power absorbed by a rotary drum seed treatment equipment can be performed. Considering that one kilogram of corn seeds contains over 3000 seeds, the calculation of power absorbed using the DEM method can only be done with the aid of computing equipment.

$$P(t) = \sum_{i}^{N} m_i g l_i(t) \cos\varphi_i(t) w_i(t) = \sum_{i}^{N} m_i g v_i(t) \cos\varphi_i(t) = \sum_{i}^{N} m_i g v_{z,i}(t) \tag{4.5}$$

where: P – power absorbed by the material (W), N – total number of particles in the drum, mi – mass of particle i (kg), g – gravitational acceleration (m/s^2), li – distance between the particle's location and the center of rotation (m), φ – angle between the drum axis and distance l, wi - angular velocity of particle i (rad/s), vi – velocity of particle i (m/s), vz,i – vertical velocity of particle i (m/s).

Calculation of the power required for seed treatment equipment through vacuuming

In the process of seed treatment through vacuuming, the mechanical work required for the vacuuming of a cylindrical chamber is equivalent to lifting a column of mercury (of 760 mm) to a height equal to the height of the cylindrical chamber. For the complete evacuation of air from the vacuum-treated chamber, it is necessary to perform mechanical work determined according to the following relationship:

$$L = m \cdot g \cdot h \ (\text{kg} \cdot \text{m}^2/\text{s}^2) \tag{6.23}$$

where: L - mechanical work, m – mass of the mercury column, g – gravitational acceleration, h – height of the vacuum chamber. The power required for vacuuming the treatment chamber is calculated according to the following relationship:

$$P = L / t \ (\text{W}) \tag{6.24}$$

where: P – power, L – mechanical work, t – vacuuming time.

Calculation of the quantity of pesticides per seed

The model for determining the quantity of pesticides per seed (Table 9) consists of eight independent variables and four dependent variables. The model is based on three key components: LD_{50}, protected biomass, and the feeding percentage of the insect's body mass. LD_{50} SA represents the amount of substance administered to a group of individuals resulting in the death of half the population. Within the model, this parameter can be replaced with other values of lethal dose depending on the chosen control strategy. Protected biomass represents the mass of the plant in which the pesticide concentration has lethal values for target organisms. The feeding percentage of the insect's body mass is a factor dependent on the biological stage and climatic conditions. The final result of the model represents the quantity of pesticide transferred to the seed mass. Independent variable 3, the concentration of the treatment solution, represents the value related to the specific volume of the seeds.

Table 9. Variables of the model for determining the quantity of pesticide per seed (Fătu et al., 2021)

1.	MMB – mass of 1000 seeds (g)	Determined by counting and weighing
2.	U – seed moisture (%)	Determined using a thermobalance
3.	C – concentration of the active substance in the treatment product (%)	Manufacturer's specifications
4.	MSU – dry mass of a seed (g)	MSU = MMB·(100 – U) /100000
5.	DL_{50} S.A. insects (μg/g insects)	Value from the specialized literature
6.	Bp – protected plant biomass (g)	User-specified
7.	Bi – insect biomass (g)	Determined by weighing
8.	Phi - feeding percentage of the insect's body mass (%)	Experimental value
9.	SA1s – active substance per seed (μg)	$SA1s = (Bp \cdot DL_{50} \cdot Bi \cdot 100) / Bi \cdot Phi$ $SA1s = Bp \cdot DL_{50} \cdot 100 / Phi$
10.	SAts – active substance per ton of seeds (g)	Sats = 1000·SA1s/MMB
11.	PPPts – product per ton of seeds	PPPts = (Sats·100)/(100-C)
12.	Quantity of A.S/Ha (g)	

Calculation of the volume of water absorbed in the seed treatment process through vacuuming

By vacuuming the air from inside the seeds immersed in liquid, there is a forced displacement of air with liquid during pressurization from 100 Pa to 100 kPa. The transport of water inside the seeds in this case is similar to the transport through a capillary bundle and can be approximated using the Poiseuille's equation:

$$Q = \frac{\pi r^4 \Delta P}{8 \mu L} \tag{5.16}$$

where: Q – flow rate (m/s); π – constant; ΔP – difference between the initial and final pressure (Pa); μ - viscosity coefficient of the absorbed liquid (kg/ms); r - radius of the tube or capillary bundle (m).

The water sorption phenomenon is positively influenced by the high porosity of the seeds (the diameter of cell cavities and intercellular capillaries) and the pressure difference between atmospheric pressure and the pressure reached during vacuuming. The fluid transport speed inside the seed is reduced by the increased viscosity of the liquid and frictional forces in vessels. Because the porous structure of the seed allows simultaneous transport through multiple capillaries, frictional force can be neglected.

Calculation of material balance and energy balance for seed treatments

In the process of treating seeds with plant protection products, mass transfer involves adding a quantity to the seed mass. Depending on the treatment method, the amount of plant protection product varies as follows:
- coating up to 20%;
- treatment with dry powders 5-6%;
- treatment with liquids 2%;
- treatment with semi-moist powders 0.3-0.8%;
- treatment with volatile substances (fumigation) 0.25-0.35 g/ton.

Treating seeds using vacuum allows for a mass transfer of about 2.5% of the seed mass for plant species cultivated for grains.

The energy transfer into the seed mass has a maximum consumption value of 1 Wh/kg of seeds. Depending on the equipment used and the treatment method, the energy absorbed by seeds is divided into two components: kinetic energy and thermal energy. In rotary equipment, the absorption of kinetic energy predominates, while in static equipment, thermal energy is predominant. Treating seeds using vacuum is characterized by negligible absorption of kinetic energy. However, due to the rarefied atmosphere in the treatment chamber behaving like an adiabatic insulator, the amount of absorbed energy contributes entirely to raising the temperature or activating chemical reactions inside the seed.

4.13. Design and implementation of seed treatment equipment through vacuuming

One of the vacuum seed treatment equipment used in experimental research (fig. 17.) was conceived, designed, and implemented within the SMART project, MySMIS Code 15734, at the research unit INMA Bucharest. It consists of 14 components: a food-grade stainless steel treatment chamber (diameter 24 cm, height 24 cm, thickness 1.5 mm); two plexiglass covers, with thicknesses of 10 mm and 20 mm; a 220 V, 400 W electric heating mantle; an electronic thermostat with a 1% precision NTC 10 k thermistor temperature sensor; a siliconized rubber gasket for the cover; a three-way distributor; two analog pressure gauges; four valves; two protection filters; two borosilicate glass vessels (1 L and 2 L) with GL 45 thread; two covers with GL 45 thread, with three and two ways, respectively; a vacuum pump V-i120 sv, 220 V, flow rate 51 l/min, 0.1 mm Hg column.

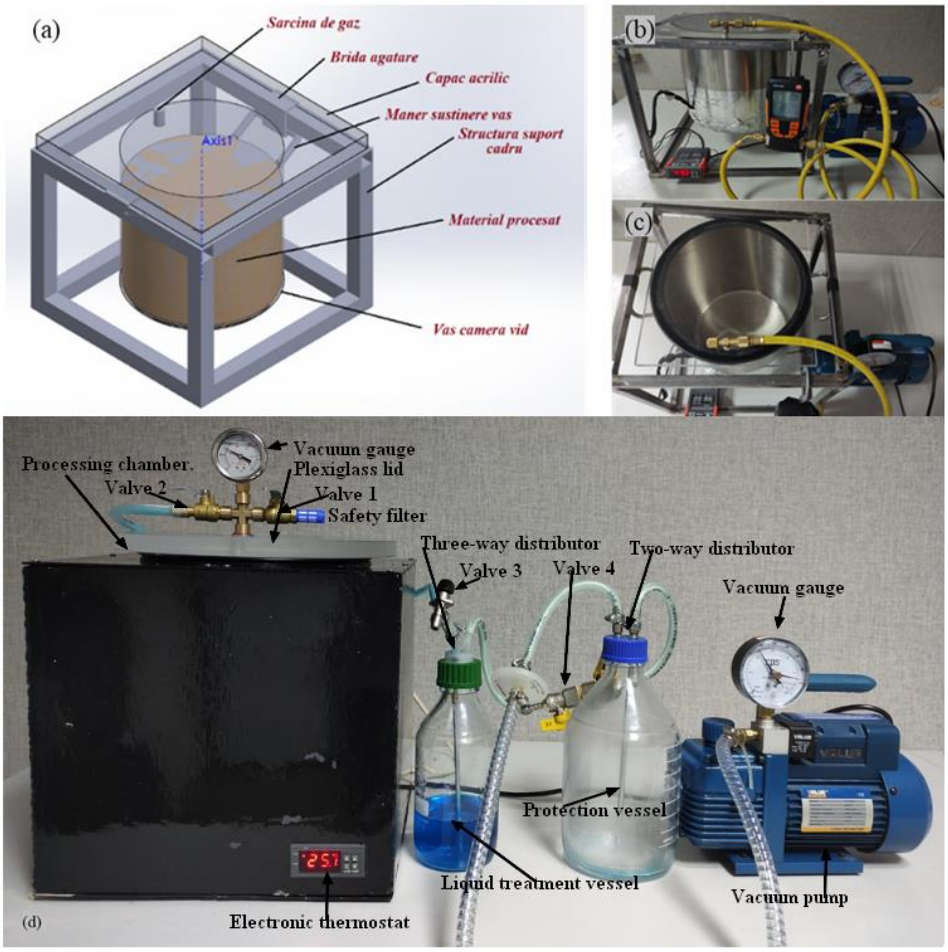


Fig.17. Seed treatment equipment by vacuuming: a) 3D geometric model; b) front view; c) top view; d) aspects during testing.

The seed treatment process using vacuum involves a series of activities in which the treatment liquid and seeds are degassed, the liquid is transported based on pressure differences, the liquid is absorbed into the seeds through diffusion in a vacuum, any unused liquid is recovered, and optionally, the seeds are dehydrated:

- load the one-liter vessel with the treatment solution.
- load the treatment chamber with seeds.
- close valve 1 (chamber pressurization).
- open valve 2 (on the lid).
- close valve 3 on vessel 1.
- open valve 4 for access to the vacuum pump.
- start the vacuum pump and let it operate until the vacuum reaches the desired value in the treatment chamber.
- close valve 4.

- open valve 3, allowing the liquid to be transported from vessel 1 into the treatment chamber until the seeds are immersed in the liquid, and the pressure in the treatment chamber equals atmospheric pressure.
- close valve 3.
- allow the seeds to rest for 6-10 minutes to allow the liquid to be absorbed into the porous structure of the seeds.
- open valve 4 and start the vacuum pump until any unused liquid is recovered.
- close valve 2 and open valve 1 to pressurize the treatment chamber.
- evacuate the treated seeds.
- optionally, the heating system can be used simultaneously with vacuuming for the dehydration of seeds for long-term preservation.

4.14. Experimental determination of the specific absorption volume of pesticides by seeds treated through vacuuming

The *specific absorption volume* or technologically can be defined as the volume of water that seeds can capture after being immersed in water following the vacuuming of air from the pores of the seeds. The protocol for determining the volume of substance absorbed by seeds involved loading 25 grams of seeds into the treatment device, immersing them in distilled water in a Berzelius glass, and placing them in the vacuum chamber (fig.18).

Fig.18. Treatment device loaded with 25 grams of seeds

After vacuuming at different levels of internal pressure, the chamber was pressurized to atmospheric pressure, and the seeds were kept immersed for six minutes to allow water to diffuse inside the seeds. Finally, the seeds were blotted with filter paper to remove excess water and weighed.

The vacuum percentage was determined according to the following relationship:

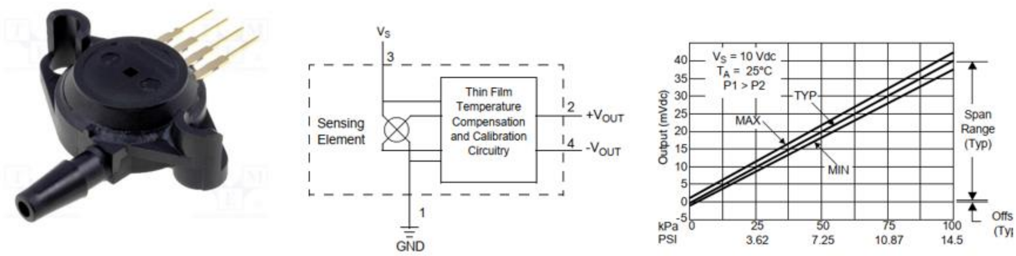
$$V_{ac} = 100 - (p_v / p_{atm})\ 100 \quad (\%) \tag{6.15}$$

where: Vac – the percentage of vacuum in the treatment chamber; p_v – the absolute pressure of the air in the treatment chamber; p_{atm} – the atmospheric air pressure under standard conditions.

The indication of reaching different percentages of vacuum in the chamber was achieved by measuring the output voltage of the signal generated by the MPX2100AP pressure sensor, whose sensitivity was 0.40 mV/kPa when supplied with 10 Vdc according to the following relationship.:

$$p_i = mV_{cc} \cdot 100000 / 40 \tag{6.16}$$

where: pi – the pressure inside the chamber (Pa); mVcc – the value of the direct current voltage measured across terminals 2 and 4 of the sensor (fig.19).



(a) (b) (c)

Fig.18. Thermally compensated and calibrated absolute pressure sensor: (a) construction form 344B, (b) electrical schematic, (c) linear pressure/output voltage characteristic

The specific volume was calculated by the difference between the seed mass before treatment and after treatment, relative to the density of the treatment liquid:

$$V_s = (M_{is} - M_{fs}) / \rho_s \tag{6.17}$$

where: Vs – specific volume (ml/100g seeds), M_{is} – initial mass of seeds (g), M_{fs} – final mass of seeds (g), ρ_s – density of the treatment liquid (g/cm^3).

The amount of water absorbed through the vacuuming process was calculated by the difference between the total amount of absorbed water and the amount of water absorbed at atmospheric pressure.

The results obtained for the specific absorption volumes of the five cereal species in the experiments were lower than the internal porosity of the seeds, except for oats. The coefficient of determination between the vacuum pressure before sorption and the amount of absorbed liquid had values ranging from 0.88 to 0.97. These regression coefficient values demonstrated a strong correlation between the air evacuated from inside the seeds and the volume of absorbed water. Figures 18-22 present the quantities of liquid absorbed by the five cereal species for grains depending on the vacuuming pressure.

In conclusion, experimental research on the sorption of distilled water inside the seeds after evacuating the air through vacuuming demonstrated that the specific volume of absorption is generally smaller than the volume of internal seed porosity.

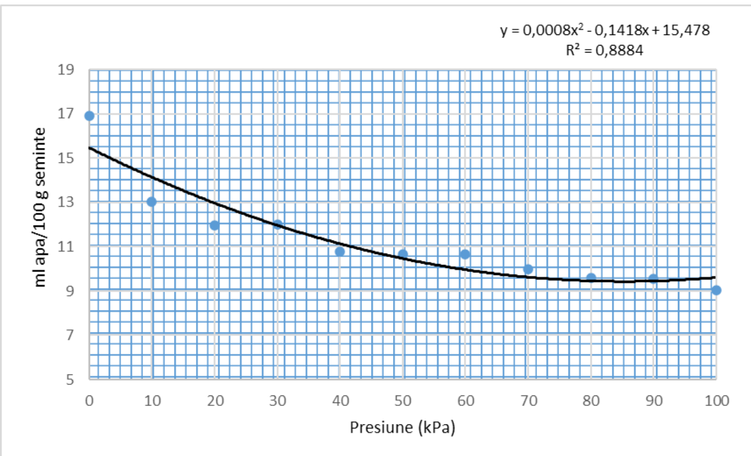


Fig.19. The specific volume of corn seeds

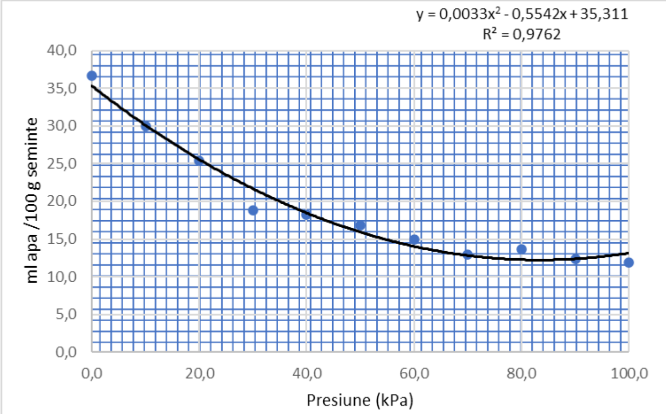


Fig.20. The *specific* volume of water absorbed by barley seeds

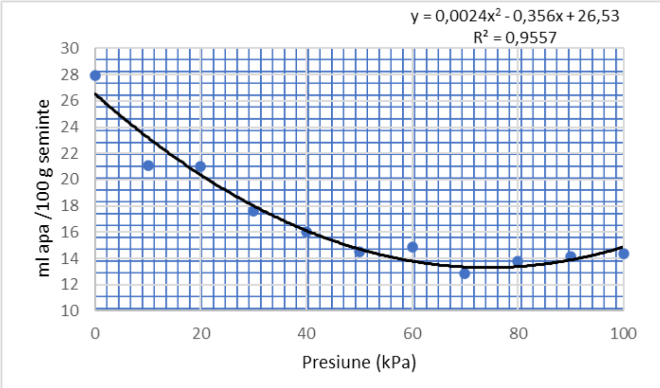


Fig.21. The specific volume of rye seeds

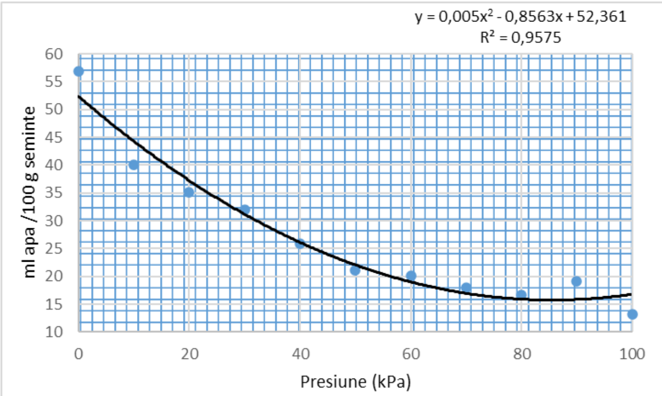


Fig.22. The specific volume of oat seeds

4.15. Experimental determination of the specific volume of pesticide adsorption by seeds treated through atmospheric pressure immersion

This study aimed to evaluate the deposition of methiocarb from the aqueous solution into the mass of corn seeds in a batch-type adsorption system. The influence of flow rate, initial solution concentration, and seed bed length on the adsorption of methiocarb by the seed mass in a fixed-bed column in a laboratory-scale setup was investigated. Additionally, a model based on a neuro-fuzzy inference system was developed to estimate the adsorption of the active substance based on process performance parameters.

In fig. 23, *the influence of the initial concentration of the active substance in the treatment solution* on the adsorption process is presented at a constant flow rate of 12 ml/min and a corn seed bed length in the adsorption column of 200 mm. It shows that the adsorption percentage decreases from 89.0% to 77.0% with an increase in the initial concentration of methiocarb from 23 ppm to 86 ppm, even though the adsorption density increases. Under similar experimental conditions for the adsorption of a crystal violet dye on a tamarind seed powder, Patel and Vashi (2010) found in their study that the given adsorbent mass can only adsorb a fixed amount of the adsorbate. Consequently, as the solution becomes more concentrated, the decrease in the adsorption of methiocarb molecules may be due to the dissolution of adsorbate species and changes in pore size, but further evidence is needed regarding the role of diffusion between particles in determining the adsorption rate. [26, 27].

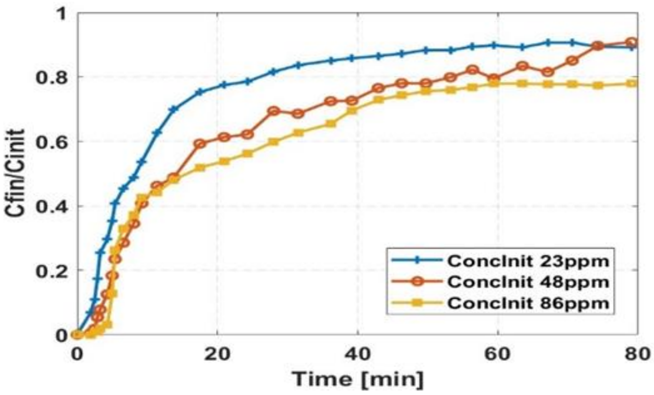


Fig.23. The influence of the initial concentration of the solution on the adsorption process

The influence of the solution flow rate on the adsorption process was studied under the conditions of a seed bed with a length of 150 mm and an initial concentration of the active substance, metiocarb, at 48 ppm. In Fig. 24, the results of the adsorption curves obtained for three different values of the solution inflow rate, namely 6, 9, and 12 ml/min, are presented. As can be observed from the data summary table (Table 10), lines 4-6, doubling the solution flow rate from 6 to 12 mm/min leads to a relatively small decrease in the adsorption efficiency, from 89.1% to 84.5%, mainly due to the fact that at higher flow rate values, the dissolved metiocarb particles have much less time to diffuse into the free spaces between seeds or into the pores of the seeds.

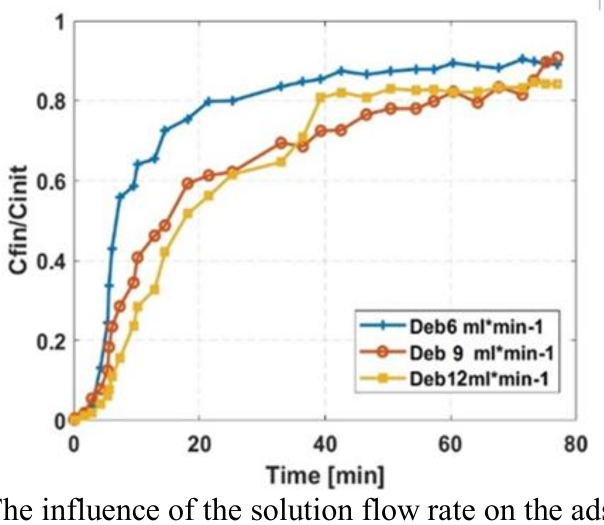


Fig. 24. The influence of the solution flow rate on the adsorption process

Table 10. Experimental results obtained under process conditions (T = 25 °C and pH = 5,7)

No	L (mm)	Q, (ml min⁻¹)	C_{init}, (mg /l)	Time, (min)	q_{total}, (mg)	q_{eq} (mg/g)	η_e (%)	η_{sn} (%)	Ɛ (%)
1	100	6	23	80	$1,73.10^{-03}$	$7,08.10^{-06}$	94,32	97,6	3,48
2	150	6	23	80	$1,44.10^{-03}$	$5,87.10^{-06}$	89	92,3	3,71
3	200	6	23	80	$1,69.10^{-03}$	$6,91.10^{-06}$	83,4	88,5	6,12
4	150	6	48	80	$4,30.10^{-03}$	$1,76.10^{-05}$	89,1	91,6	2,81
5	150	9	48	80	$6,20.10^{-03}$	$1,69.10^{-05}$	85,8	88,7	3,38
6	150	12	48	80	$7,25.10^{-03}$	$1,48.10^{-05}$	84,26	87,5	3,85
7	200	12	23	80	$4,04.10^{-03}$	$8,27.10^{-06}$	89,1	92,4	3,70
8	200	12	48	80	$8,89.10^{-03}$	$1,82.10^{-05}$	80,7	83,6	3,59
9	200	12	86	80	$1,899.10^{-6}$	$3,84.10^{-05}$	77	80,5	4,55

The Influence of Bed Length. Bed length in adsorption refers to the distance covered by an adsorbent material in an adsorption process. This length can significantly affect the efficiency and performance of an adsorption system and may vary depending on several factors.

Figure 25 illustrates a graph showing the variation of the adsorbate concentration to initial solution concentration ratio (C_{fin}/C_{init}) obtained for the adsorption of methiocarb on corn seeds at different fixed bed lengths of 100, 150, and 200 mm (corresponding to masses of 245, 367, and 500 g of adsorbent), at a constant flow rate of 6 ml/min and an inlet concentration of 23 mg/l.

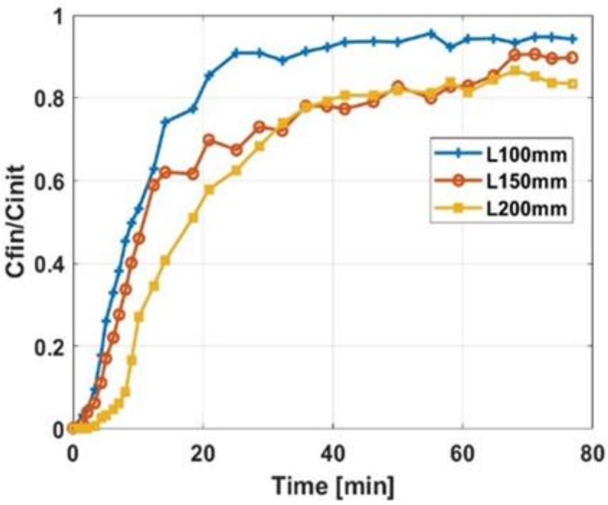


Fig.25. Influence of adsorption bed length on the adsorption process

If the adsorption bed is too short, it may not provide enough time for substances to be adsorbed onto the adsorbent's surface in significant amounts. This can lead to reduced efficiency in the adsorption process. On the other hand, a bed that is too long can result in unnecessary loss of time and energy. It is interesting and easy to observe from both Figure 25 and Table 10 that the adsorption efficiency of methiocarb decreased with the increase in the seed bed length. The efficiency dropped from 94.32% for a bed length of 100 mm to 89% for a bed length of 150 mm and further to 83.4% for a bed length of 200 mm. Consequently, a shorter seed bed length favored the adsorption capacity of methiocarb, in line with findings reported by other authors [28, 29, 30, 31, 32, 33, 34].

In conclusion, the research investigated the adsorption capacity of the methiocarb substance by corn seeds to obtain an alternative treatment with low cost and efficient batch operation. Since a portion of the pore spaces between seeds is inaccessible to the treatment solution, the adsorption of the active substance may be very limited. Thus, the seeds may have a lower level of protection against pests.

4.16. Numeric and experimental analysis of the vacuum seed degassing system

Vacuum System

To achieve the necessary vacuum inside the chamber, a low-pressure mechanical pump was utilized. The vacuum pump is a rotary vane mechanical pump by Value (2020), with a maximum rotation speed of 1440 rpm, a power of 200 W, a flow rate at 220 V/50 Hz of 42 l/min or 1.5 CFM. The pump has the necessary capabilities for high vacuum, includes a built-in manometer to measure gross vacuum, and can reach pressures ranging from 101325 Pa to 20 Pa. To measure medium and high vacuum pressures within the chamber, a portable Testo 552 device was used, as shown in fig. 26.

Fig.26. Portable device measuring vacuum pressure

Determining the pumping time to reach the desired vacuum

a) <u>*Evacuation of the gas in a chamber under rough vacuum*</u>

In the case of evacuating the gas from a chamber under rough vacuum (without additional sources of gas or vapors), the effective pumping speed S_{eff} of a pump-chamber assembly depends only on the required pressure p, the volume V of the container, and the pumping time t.

With a constant pumping speed S_{eff}, assuming that the maximum pressure reached with the pump model is such that $p_{fin} << p$, the pressure drop p(t) over time in a vacuum chamber is given by the first-order differential equation., [35]:

$$-\frac{dp}{dt} = \frac{S_{eff}}{V} \cdot p \qquad (7.6)$$

Starting with the value of 1013 mbar at time t = 0, the effective pumping speed is calculated as a function of pumping time t from equation (7.6), as follows:

$$\frac{\ln p}{1013} = -\frac{S_{eff}}{V} \cdot t \qquad (7.7)$$

$$\int_{1013}^{p} \frac{dp}{p} = -\frac{S_{eff}}{V} \cdot t \tag{7.7a}$$

$$S_{eff} = \frac{V}{t} \cdot \frac{\ln 1013}{p} = \frac{V}{t} \cdot 2{,}3 \cdot \frac{\log 1013}{p} \tag{7.7b}$$

By introducing the dimensionless pressure factor σ

$$\sigma = \frac{\ln 1013}{p} = 2{,}3 \cdot \frac{\log 1013}{p} \tag{7.8}$$

The relationship between the effective pumping speed S_{eff} and the pumping time t is given by the equation:

$$S_{eff} = \frac{V}{t} \cdot \sigma \tag{7.9}$$

The ratio V/S_{eff} is generally referred to as a time constant τ. Thus, the pumping time of a vacuum chamber from atmospheric pressure to a pressure p is given by:

$$t = \tau \cdot \sigma \tag{7.10}$$

with $\tau = \frac{V}{S_{eff}}$ și $\sigma = \frac{ln^{1013}}{p}$.

The dependence of the σ factor on the desired pressure is presented in Fig. 27. It should be noted that the pumping speed of single-stage rotary vane pumps and rotary piston pumps drops below 10 mbar with gas ballast and below 1 mbar without gas ballast.

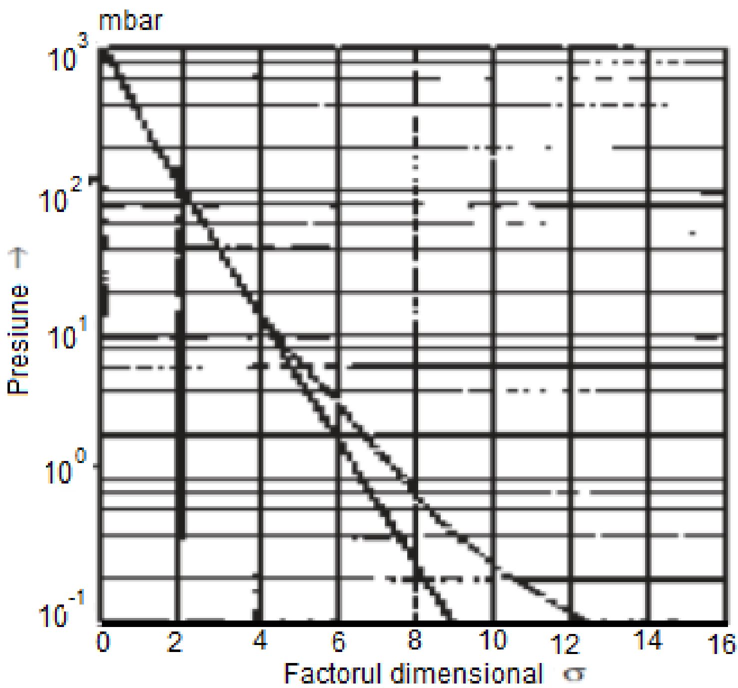


Fig.27. The dependence of the dimensionless factor σ for calculating the pumping time t. The dashed line appears for single-stage pumps when the pumping speed drops below 10 mbar., [36]

b) Evacuation of gas from a chamber in medium vacuum conditions

In the rough vacuum regime, the volume of the vessel is decisive for the time involved in the pumping process. In the high and ultra-high vacuum regions, however, gas desorption from the walls plays a significant role.

In the medium vacuum region, the pumping process is influenced by both quantities. Moreover, in the medium vacuum region, especially with rotary pumps, the maximum pressure that can be achieved is no longer negligible. If it is known that the amount of gas entering the chamber is at a rate Q (in millibar liters per second) from the desorption of gas from the walls and leaks, the differential equation (7.6) for the pumping process becomes, [35]:

$$dp/dt = -(S\_eff\,(p - p\_fin) - Q\dashv)/V \tag{7.11}$$

The integration of this equation leads to:

$$\frac{t = \frac{V}{S_{eff}} \ln\left((p_0 - p_{fin}) - \frac{Q}{S_{eff}}\right)}{(p_0 - p_{fin}) - \frac{Q}{S_{eff}}} \tag{7.12}$$

where: p_0 - the pressure at the beginning of the pumping process; p_{fin} - the desired pressure.

Unlike equation (7.7, b), this equation does not provide a definition for S_{eff}, hence the effective pumping speed for a known gas throughput cannot be determined from the pressure/time curve without additional information.

Therefore, in practice, the following steps are taken:

a) the pumping speed is calculated from equation (7.7) as a result of the volume of the gas release-free chamber and the desired pumping time;

b) the ratio between the gas release speed and this pumping speed is found.

This ratio should be lower than the required pressure. For safety, its value should be about ten times smaller. If this condition is not met, a pump with a correspondingly higher pumping speed must be chosen.

c) Evacuation of the gas from a chamber in the high vacuum regime

In cases where the pumping process is dominated by residual gas, pumping in a high vacuum region can be described by the relationship (7.8):

$$p = p_0 \cdot \exp\left[-\frac{\left(\frac{S_{eff}}{V_{tot}}\right)}{t}\right] \tag{7.13}$$

where: p - pressure after time t; p_0 - pressure at time t = 0; S_{eff} - effective pumping speed; V_{tot} - total volume of the system.

However, by far the most significant uncertainty associated with pump performance, pressure measurements, flow rates, and external leaks is due to gas release. Release rates can easily vary by many orders of magnitude depending on the history and material of a surface, its treatment, humidity, temperature, and exposure time to vacuum. Because it usually asymptotically approaches the final pressure of a system, even small changes in gas loads result in large differences in evacuation times.

The vacuum analysis that helped select a rough vacuum pump was based solely on the relationship between pressure and the time until that pressure was reached.

Relevant assumptions for such an analysis are:

- the system has no leaks;
- the pumps are 100% efficient;
- nothing will vaporize in the chamber.

Theoretical simulation of pumping time was performed using a VBA module in an Excel spreadsheet based on mathematical models containing design parameters and parameters characterizing the initial conditions (D, H, V, pfin, σ, and Seff). Figure 28 shows the VBA macro interface with the simulation of pressure variation over time.

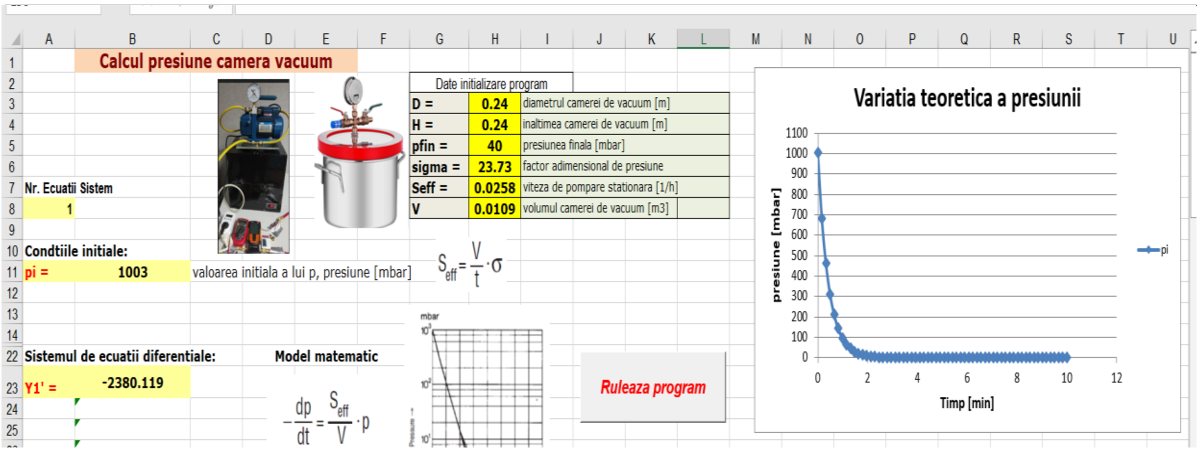


Fig.28. The graph of “gas load pressure” versus time

According to the statistical analysis between theoretical data from numerical simulation (fig.29) and experimental data (fig.30), it can be concluded that the vacuum process can be mathematically approximated with very small error, as the calculated t value (t-test) is smaller than the corresponding critical value

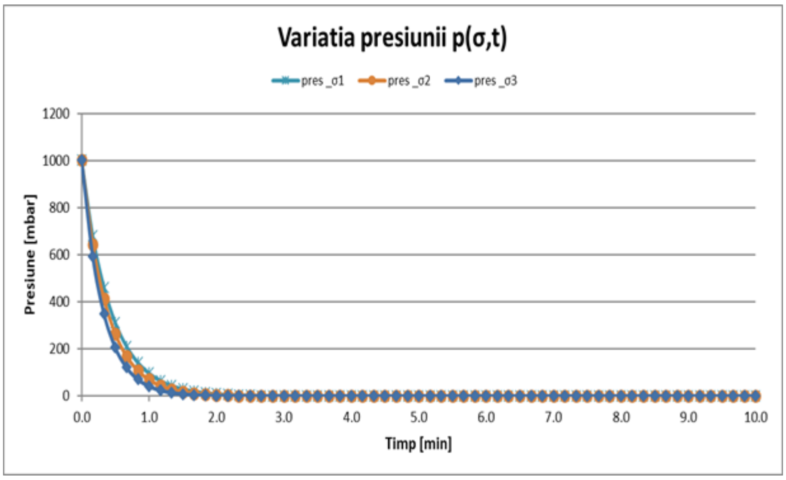


Fig. 29. Numerical simulation of pressure variation over vacuuming time

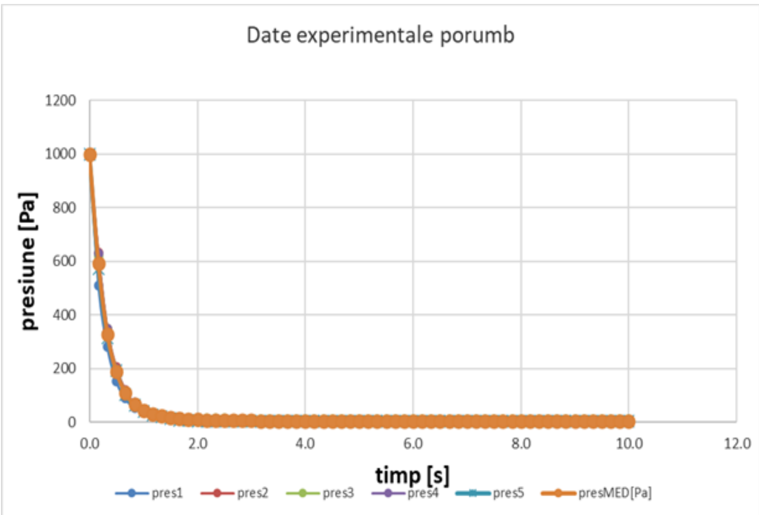


Fig.30. Experimental data of pressure variation over vacuuming time

Structural dynamic analysis with finite element method (FEA)

To investigate the structural dynamics of the system in response to various loads, a finite element analysis (FEA) is conducted. In this application, it is necessary to determine force-displacement characteristics, functional constraints, and load-carrying capacities of the vacuum chamber vessel, considering it is made of stainless steel 316, and the cap is made of acrylic plastic. The entire process of dynamic analysis is carried out in the following steps: the first step of structural dynamic analysis is the selection of material and its data; various materials can be tested under vacuum pressure, but the most commonly used types are metals, plastics, and their composites [37]. The ANSYS material library provided the characteristic values of materials, while the temperature and other experimental conditions were mentioned as described below. The selected material for the chamber cap is acrylic plexiglass with a density of 1.185 x 10^-6 kg/mm³, a Poisson's ratio of 0.3952, Young's modulus of 3225 MPa, stiffness modulus of 5182.9 MPa, shear modulus of 1167.9 MPa, and the vessel is made of stainless steel 316 with a density of 7.985 x 10^-6 kg/mm³, a Poisson's ratio of 0.25, Young's modulus of 1.95 x 10^5 MPa, stiffness modulus of 1.3 x 10^5 MPa, shear modulus of 78000 MPa. The 3D geometry of both the chamber vessel and the cap was created in SolidWorks 2022 based on the dimensions of the experimental physical model. The dimensions for the cylindrical chamber vessel are 240x240x3 mm (diameter, height, wall thickness), and for the cap are 240 x15 mm (diameter, cap thickness). Modeling of contact-type connections between the vessel and the cap was automatically done using the Augmented Lagrange method for solving the nonlinear model of connections without friction. The discretization was performed automatically with implicit parameters for both the vessel and the cap. Using the adaptive mesh sizing method resulted in a total of 10546 nodes and 4572 elements (8968 nodes and 4363 elements for the vessel; 1578 nodes and 209 elements for the cap).

Total Deformation. Table 11 synthetically presents the deformation values of the test obtained through structural dynamic analysis in relation to the vacuum pressure in the test experiment. The visual representation shown in Fig. 31 represents the maximum deformation produced at the bottom of the chamber as well as the deformation of the cap.

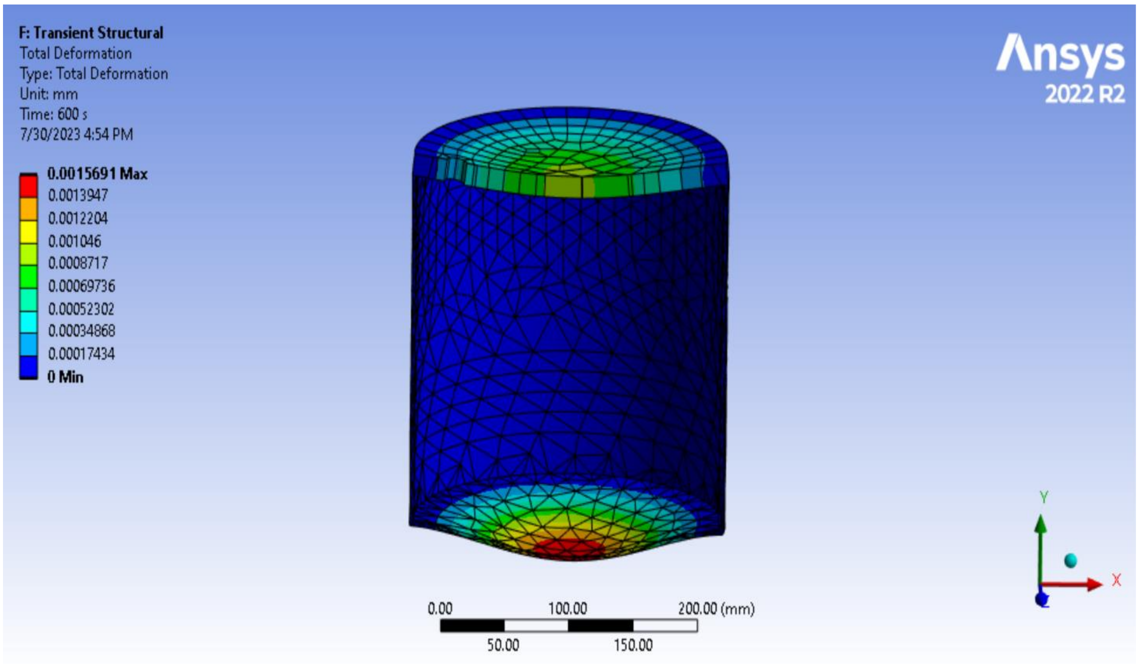


Fig. 31. The values of deformations occurring on the wall of the vacuum chamber and the cap

The color scheme presented in fig.31 explains the deformation caused by the pressure load. The maximum deformation of 0.001569 mm was observed in the middle part of the vessel's bottom and decreases significantly towards the exterior.

In conclusion, the dynamic and structural finite element analysis highlighted that the dimensions of the treatment chamber are correctly chosen to withstand the mechanical stresses due to the pressure difference.

Table 11. The total deformations caused by the vacuum pressure through ANSYS simulations

Time (s)	Minimum (mm)	Maximum (mm)	Mean (mm)
0	0	$4{,}5\cdot10^{-1}$	$2{,}82\cdot10^{-2}$
10		0,35917	$2{,}24\cdot10^{-2}$
20		0,28578	$1{,}78\cdot10^{-2}$
30		0,22737	$1{,}42\cdot10^{-2}$
40		0,18093	$1{,}13\cdot10^{-2}$
50		0,14403	$8{,}97\cdot10^{-2}$
....		...	
560		$1{,}57\cdot10^{-3}$	$1{,}03\cdot10^{-4}$
570		$1{,}57\cdot10^{-3}$	$1{,}03\cdot10^{-4}$
580		$1{,}57\cdot10^{-3}$	$1{,}03\cdot10^{-4}$
590		$1{,}57\cdot10^{-3}$	$1{,}03\cdot10^{-4}$
600		$1{,}57\cdot10^{-3}$	$1{,}03\cdot10^{-4}$

4.17. Validation of the seed treatment technology by vacuumization using bioassays that characterize seed vigor

The validation of the seed treatment method was carried out by treating corn seeds with the insecticide imidacloprid using the equipment developed in the context of the theme through a bioassay evaluating germination and seedling growth in the early vegetative stages. Table 12 presents the concentrations of the treatment doses, and Figure 32 shows the effects of the treatment on the number of germinated seeds and root growth

Table 12. The experimental variations for testing the active substance at different concentrations on corn seeds (treatment with imidacloprid))

Treatment	V1-250 ppm	V2-500 ppm	V3- 1000 ppm	V4-2000 ppm	V5-3000 ppm	V6-6000 ppm	V7-0 ppm Control
Dose, ppm	250	500	1000	2000	3000	6000	0
SA, mg	1,44	2,89	5,78	11,56	17,34	34,67	0
Mixture volume, µl	28,89	57,79	115,58	231,15	346,73	693,46	0
water volume, µl	764,15	735,26	677,47	561,89	446,32	99,58	793,05
AS/kg seeds, g/ton	250	500	1000	2000	3000	6000	0
µg AS/seed	57,79	115,57	231,15	462,30	693,46	1386,92	0

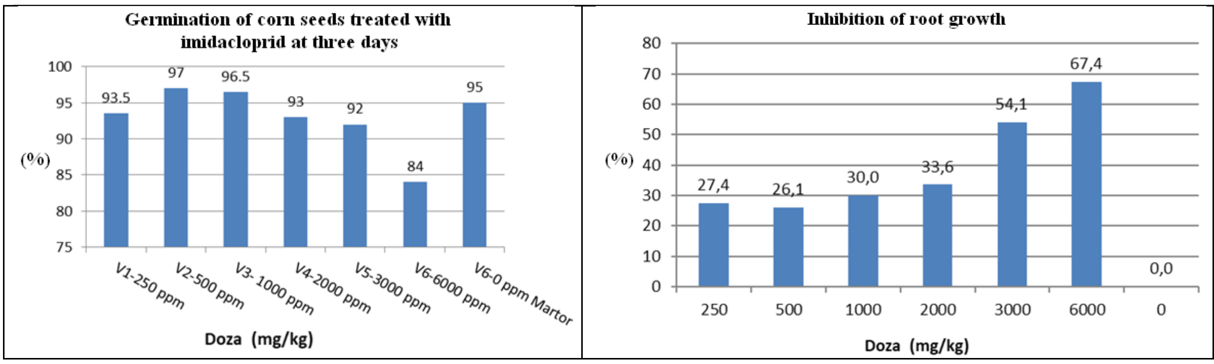


Fig.32. The effect of imidacloprid on germination and radicle growth of corn germs

In conclusion, the vacuum treatment of corn seeds with imidacloprid ensured a sufficient transfer of the active substance to slow down the growth of embryos during the germination period. The vacuum treatment process covered a range of insecticide doses from 250 grams to one ton of seeds up to 6 kg per ton of seeds. Currently, a maximum of 3.6 kg of insecticide per ton of seeds is commonly used to combat corn pests.

5. Final conclusions

Cereal crops cover an area of over five million hectares in Romania, producing nearly nineteen million tons of seeds according to statistical data published in 2022 by the National Institute of Statistics. Over the past 20 years, the average quantity of pesticides used per hectare has been below one kilogram. With an average cultivated area of eight million hectares in Romania, an annual pesticide consumption of 5.6 million kilograms can be estimated. The average pesticide consumption in the country, at 0.7 kg/ha, is moderate compared to some countries like China or Brazil, where consumption can reach 10-15 kg/ha. The quantity of insecticides needed for seed treatment for establishing grain crops is approximately 450 tons of active substance. Allocating this quantity to the cultivated agricultural area for grain plants results in an average consumption of 90 g/ha.

Data from some published works demonstrate that at least 20% of the treated product mass remains in the soil, and another 20% is used unnecessarily. Although the quantity of insecticides used for seed treatment is small, due to a half-life as long as 1000 days, there is a multi-year accumulation leading to long-term environmental pollution.

Considering that non-polluting insecticides based on biological substances do not yet have the same effectiveness as synthetic substances, and the European Commission's strategy outlined in the "Green Deal" requires a halving of pesticide consumption by 2030, one option is to optimize seed treatment for current active substances.

To address this challenge, the research theme aimed to conduct studies with a new model of seed treatment, allowing protective active substances to become an integrated part of the seed's internal structure, providing the embryo with a competitive advantage for internal and external nutritional resources.

The main objective of the research was to identify and describe the physical and biological phenomena that occur during seed treatment using vacuum as a method to accelerate the transfer of active substances into the porous matrix of the seeds.

Secondary objectives were related to the design, development, and implementation of a seed treatment device using vacuum and defining the working parameters of the equipment through physical-mathematical simulations and laboratory experiments.

The proposed vacuum seed treatment technique is an improved version of the immersion seed treatment technique in liquid solutions and borrows characteristics from the technique of treating seeds with cold plasma. Immersion seed treatment has the disadvantage of requiring a long immersion time, during which phenomena of embryo suffocation and loss of mineral salts through diffusion into the treatment liquid can occur, leading to a decrease in seed vigor.

Vacuum seed treatment involves replacing the air from the internal porous structures of the seeds with the liquid treatment product. The treatment process takes place in two consecutive stages: in the first stage, air is evacuated from the immersed seeds, and in the second stage, liquid transfer occurs inside the seeds as the liquid pressure increases from 200 Pa to 100 kPa. The amount of transferred liquid can be controlled in the first stage by achieving an appropriate vacuum percentage before pressurization or in the second stage by interrupting imbibition after a certain time after pressurization.

To achieve the proposed objectives in the experimental research, two vacuum seed treatment stands and one stand for seed treatment by immersion at atmospheric pressure were built. The first seed treatment stand has a treatment chamber volume of 8.16 liters and is equipped with a system of pipes and valves that allow the chamber to be evacuated, the introduction of the treatment liquid, and the evacuation of excess using the pressure difference created by a vacuum pump. The second stand is thermally insulated, has a treatment chamber volume of 10.85 liters, and is equipped with an automated electric heating system. The third stand has a treatment chamber volume of 0.7 liters, and the treatment liquid is continuously circulated using a peristaltic pump.

Final conclusions on theoretical and experimental research

Treating seeds before planting is a crucial technological process, as the success of initiating crops in the early stages of vegetation depends on the quality of treated seeds. From an ecological standpoint, seed treatment has the lowest impact on the environment, utilizing the smallest amount of pesticide per hectare (e.g., 90 grams in seed treatment compared to 140 grams in foliar treatment with imidacloprid). Understanding the physical-mechanical and technological properties of seeds is the first step in defining the specific volume that can be used as a loading space for the product. From an economic efficiency perspective, seed treatment with plant protection products uses doses that strike a balance between recorded plant losses and pest density. Thus, in the case of low pressure exerted by pests on plants, smaller doses than effective ones can be used. The performance of seed treatment depends equally on the effectiveness of the product used and the quality of the seed treatment process.

In theoretical research, most types of existing equipment, either physically or in published patents, were identified. From the analysis of the working process of seed treatment equipment, it was observed that the kinetic energy for homogenization is distributed towards both seeds and the treatment product. On average, the power absorbed by the equipment for homogenization was around 1 Wh per kilogram of seeds.

Homogenizing seeds with the treatment product is achieved through rotational, rolling, lifting, and dropping movements of the seeds inside the working space of the equipment. These movements are accompanied by friction and collision phenomena that can induce negative changes in the mechanical integrity and morphological structure of the seeds. High-performance equipment, used by companies specialized in seed treatment, is based on working processes where the kinetic energy required for homogenization is predominantly directed towards the treatment product. Using this technique, seeds better retain their mechanical integrity.

Sometimes, adding water to the homogenization process shortens the period of mechanical stress on the seeds, facilitates mass transfer, and reduces energy consumption but has a negative effect on the storage period of seeds after treatment. The morphological and physiological properties of seeds impose limits on the speed of executing the seed treatment process. The more fragile the morphological protective structure of the seeds, the more the kinetic moment on the seeds must be reduced.

The study of technical-scientific literature highlighted that analysis models using the discrete element method can establish kinetics and calculate individually absorbed energy for each seed in a batch. Therefore, using these models can simulate the treatment process in rotationally moving equipment, necessary for designing and determining optimal working parameters.

In experimental research, experiments were conducted demonstrating that seed treatments using vacuum technology can achieve better or at least similar results to rotary homogenization concerning the quantity of pesticides added to the seed mass. Bioassays showed that the vacuum process has no negative impact on the health and mechanical integrity of the seeds. The experimental study of the sorption phenomenon of liquid pesticides revealed that, during seed treatment using vacuum technology, absorption is the main form through which mass transfer occurs. By adsorption, a mass of pesticides on the order of milligrams can be transferred to 100 grams of seeds, while absorption can achieve a transfer on the order of grams.

Personal and original contributions of the work

Within the thesis, the hypothesis was put forward that the quantity of pesticides used in seed treatment can be reduced by developing a process that involves evacuating air from seed pores and replacing it with active substances.

To verify this hypothesis:

* Two experimental stands were created:

- the first stand with a volume of 8.16 liters was adapted for transferring the treatment liquid from the reservoir to the treatment chamber and vice versa using a single vacuum pump.
- the second stand with a volume of 10.8 liters was adapted for work at controlled temperatures.

* Throughout the research, several physical-mathematical models were created and tested, allowing simulations of sorption and mass transfer phenomena, closely aligned with observations made during experiments.

* The pesticide dose calculation model, using biological parameters related to the lethal dose, duration of protection offered, seed morphology, and technological parameters related to pressure, treatment duration, and product characteristics, represents a significant contribution to both the agricultural and environmental protection fields.

The difference between experimentally obtained values and those obtained through simulation regarding the absorption of liquid products in the seed mass was 0.3%. This fact demonstrates that the model can be successfully used to control the working process of seed treatment equipment using vacuum technology.

By using vacuum technology in the seed treatment process, a new treatment method is obtained, combining characteristics of the immersion treatment method with features of the treatment method with cold plasma under vacuum conditions.

Using this seed treatment method can theoretically achieve an eightfold reduction in the quantity of pesticides used.

Recommendations and research perspectives

Recommendations for future research are related to:

- ✓ reducing the intensity of the water adsorption phenomenon during treatment.
- ✓ the possibility of introducing the conditioning process through dehydration at low pressures and temperatures, within the tolerance limits of the seeds.

In addition, future research perspectives involving:

- ✓ the possibility of introducing substances other than pesticides into the seed mass through vacuum technology, such as nanomaterials, enzymes, vaccines, nutrients, or hormones,
- ✓ offer a reliable experimental basis for expanding the range of products used in seed treatment.

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Annexes

List of published works

Crt. nb.	Authors, year, Title of article / Scientific paper published in the conference volume / journal, publishing house, pages (from -to)
1.	George Ipate**, Viorel Fatu,** Gheorghe Voicu, 2023, Experimental and numerical research on adsorption of measurol solution in fixed-bed corn seed treatment, U.P.B. Sci. Bull., Series D, (acceptat pentru publicare)
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Curriculum vitae Europass	
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Professional experience	
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Period	2019-present
Position or position held	Researcher in applied electronics
Main activities and responsibilities	Calibration of agrometeorological sensors
Name and address of employer	S.C. Syswin Solutions S.R.L., Strada Biharia 26, Sector 1, București, ROMÂNIA
e) Perioada	2014-present
Position or position held	Scientific researcher 3 degree

Main activities and responsibilities	- carrying out efficacy tests of plant protection products - Optical and electronical microscopy - modeling areas of favorability of plant and insect species - coordination of research projects
f) Period	2009-2014
Position or position held	Scientific researcher
Main activities and responsibilities	- carrying out efficacy tests on plant protection products - Optical and electron microscopy - modeling areas of favorability of plant and insect species
Name and address of employer	I.C.D.P.P. Bucuresti
Type of activity or sector of activity	Agricultural research
g) Period	2006-2009
Position or position held	Research assistant
Main activities and responsibilities	-electronic microscopy -identification and diagnosis of viral diseases and phloemic diseases in cereals
Name and address of employer	I.C.D.P.P. Bucuresti
Type of activity or sector of activity	Agricultural research
Education and training	
h) Period	2019
Qualification / Diploma obtained	PhD student in mechanical engineering
Main activities	Contributions and researches regarding the working process of vacuum seed treatment machines
Name and type of educational institution/training provider	Faculty of Biotechnical Systems Engineering
Level in national or international classification	ISCED level 8
i) Period	2006-2008
Qualification / Diploma obtained	Master's Degree Diploma
Main disciplines studied	Modern applications of biotechnology in agriculture
Name and type of educational institution/training provider	Faculty of Biotechnologies, USAMV Bucharest;
Level in national or international classification	ISCED level 7
j) Period	2001-2006
Qualification / Diploma obtained	Bachelor's Diploma

Main disciplines studied	Plant biotechnology
Name and type of educational institution/training provider	Faculty of Biotechnologies, USAMV Bucharest;
Level in national or international classification	ISCED level 6
Period	2017
Main activities/acquired skills	Expert in accessing European structural and cohesion funds
Name and type of training provider	Expert business center SRL
k) Period	2010
Main activities/acquired skills	Genetic Engineering Course
Name and type of educational institution/training provider	Institute for Plant Protection, Julius Kuln, Darmstadt, Germany
l) Period	2009
Main activities/acquired skills	GIS ESRI Geosystems
Name and type of educational institution/training provider	ESRI, Romania

Self assessment	**Understanding**		**Speech**		**Writing**
Nivel european (*)	listening	Reading	Join the conversation	Oral speech	Written expression
English	C1	C2	C2	C2	C2
French	A2	C1	A1	A1	A1

Social competences and skills	Good adaptability in various multicultural environments acquired during experiences abroad, a focus on learning, diligence, and integrity
Organisational skills and competences	punctuality, ability to analyze tasks and responsibilities
Technical skills and competences	Easy handling of laboratory equipment and research instruments
Computer skills and competences	Current use of Microsoft Office (Word, Excel and PowerPoint) and other dedicated professional software (BioStat, GraphPad, Prism)

November 2023

FĂTU Viorel