"AUREL VLAICU" UNIVERSITY OF ARAD
FACULTY OF FOOD ENGINEERING, TOURISM AND ENVIRONMENTAL PROTECTION

SUMMARY
PhD THESIS

SCIENTIFIC COORDINATOR
Professor Chemist Hab. Florentina-Daniela Munteanu

PhD Student
Chereji C. Bianca-Denisa

ARAD
2024

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VALORIFICATION OF SHEEP WOOL WASTE

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CONTENTS

AIM OF THE WORK

The valorization of sheep wool waste holds significant potential to revolutionize the circular economy, offering a sustainable solution to waste management and resource utilization while reducing associated costs.

The process of valorizing sheep wool waste involves converting a problematic by-product into a valuable resource that can be used in various industries. This promising solution to waste management and resource utilization inspires hope for a more sustainable future.

A significant amount of agricultural waste, including sheep wool, is produced yearly, which can have serious environmental consequences if not managed correctly, such as [specific environmental consequences].

With its complex chemical composition and natural decomposability, sheep wool emerges as a remarkably versatile material, transcending its traditional use in textiles. Its potential applications in agriculture, biomedicine, and environmental engineering are diverse and intriguing, enhancing its value and inspiring its potential to minimize negative environmental impacts.

The use of sheep wool waste is a topic of paramount importance due to its potential to address a range of environmental issues. The urgency of developing sustainable waste management strategies is crucial and immediate to minimize the impact of wool waste on the environment [6]. This urgency underscores the need for immediate action to mitigate the environmental impact of sheep wool waste.

Integrating different recovery methods improves wool waste management, increasing efficiency, reducing disposal costs, and minimizing environmental impacts. This approach also promotes a circular economy that prioritizes resource utilization and sustainability.

Therefore, this PhD thesis aims to analyze the impact of sheep wool waste on the environment and the application of spectrophotometric methods in studying the efficiency of heavy metal retention from waters contaminated with such metals.

OBJECTIVES

Sheep wool is the most popular raw material used in the textile industry. As the demand for wool is not high and production is high, huge quantities of wool end up all over the environment and become an environmental pollutant.

Sheep wool is an affordable, sustainable, and renewable natural material. Its fibres have unique and desirable properties in environmental fields, making it an excellent sorbent of environmentally hazardous contaminants.

The presence of sheep wool waste has become particularly significant. Due to the lack of rigorous wool waste management, well-defined legislation, and proper enforcement, wool waste has impacted the entire ecosystem and is a real problem that humankind is increasingly facing.

The amide, sulfhydryl, hydroxyl, and carboxyl groups in sheep wool strongly interact with heavy metal ions via valence forces. Functional groups containing nitrogen and sulphur have a strong affinity for heavy metal ions, resulting in an attraction process between them.

This is due to the presence of ionizable polar groups on the chain of amino acid residues in the protein fiber found on the surface of the wool. This group can form strong bonds with heavy metal ions. Taking cognizance of these aspects, I proposed the following objectives as the research part of this PhD thesis:

1. To evaluate the impact of sheep and wool production at global, European, and Romanian levels.
2. The efficiency of sheep wool fibers in retaining heavy metals from polluted waters.
3. Optimization of the efficiency of sheep wool filters.

CHAPTER I. THE IMPORTANCE OF SHEEP FARMING, ORIGINS AND INTRODUCTIONS

Introduction

Archaeological evidence suggests that domesticated sheep were first discovered in the Taurus Mountains of southeastern Anatolia about 10,500 years ago. This discovery indicates that this region may be the birthplace of sheep domestication.

Eventually, sheep spread into the Old World during the Neolithic period, following the paths of human migration. They followed two main routes to Europe: one across continental Europe along the Danube and the other across the Mediterranean Sea, reaching large islands such as Cyprus and Sardinia between the 9th and 6th millennia BC.

Sheep, known scientifically as *Ovis aries*, belong to the ruminant group. They have a unique dental structure, with missing upper incisor teeth and a stomach divided into four medium-sized, hoofed compartments, with the hoof split in two.

The weight of a healthy ewe varies between 45 and 100 kilograms, while a ram usually weighs between 45 and 160 kilograms. When the ewe reaches maturity, it has a total of 32 teeth. However, the number of teeth decreases to 20 during the maturing process. The front teeth are a valuable tool for determining a sheep's age, especially in the first years of life. This is because each year, a set of milk teeth is replaced by adult teeth [7].

Most sheep are categorized according to their suitability for specific purposes, such as milk production, meat, wool, skin, or a combination of these characteristics. Other characteristics used in classification include typical face color, which is usually white or black, the presence or absence of horns, tail length, and breed topography. Breeds are usually classified according to the characteristics of their wool [2].

Wool properties differ from breed to breed, with density, curvature, length, and straightness variations. Even within a single group, there are variations in wool type and quality, making wool grading an essential part of commercial fiber processing. Sheep of different breeds vary in physical dimensions, including height and weight.

CHAPTER II. WOOL FIBER MORPHOLOGY

Introduction

Sheep wool is a natural, easily accessible, durable, and renewable material. It is a natural textile fiber currently being investigated in the medical and chemical sciences.

Wool is chemically modified to improve its chemical and physical properties. These modifications are usually done on the mercapto (-SH) groups of sheep wool, which are reduced from di-sulfide bonding with alkaline reagents [10].

Sheep wool comprises 95% pure keratin, the remaining 5% comprising hydrocarbons and other substances. Between 11% - 17% of this consists of cysteine, which has a significant amount of sulfur. The diameter of wool fiber varies according to the breed of sheep, ranging from 11.5 to 47 µm. In addition, sheep wool contains 40% hydrophilic and 60% hydrophobic amino acids [10,13,14,20,24].

Wool fiber is a fibrous, keratinous protein composed of 20 different amino acids connected by a combination of hydrogen, disulfide, hydrophobic, and ionic bonds. These bonds contribute to keratin and keratin materials' mechanical strength and structural stability.

Keratin's stability, strength, and insolubility are due to the complex network structure formed by the numerous strong disulfide bonds between the thiol (-SH) groups of cysteine residues.

These bonds exist within and between keratin polypeptides, contributing to its unique properties. The structure formed by the adjacent polypeptides and sulfur-sulfur cross-links contributes to keratin's compactness.

Keratin is a fibrous protein with a significant amount of cysteine in its amino acid sequence. Its cysteine content is higher than other structural proteins, typically between 7% and 13%.

The composition of keratin is not limited to cysteine, which plays a crucial role in the formation of disulfide bonds between protein chains. It also contains significant amounts of other amino acids, such as arginine, glycine, serine, proline, glutamic acid, and aspartic acid, as well as the essential amino acids valine, leucine, and threonine [10].

CHAPTER III. SPECIAL CHARACTERISTICS AND FIELDS OF APPLICATION OF WOOL FIBER

Introduction

Because of its many advantages, sheep wool fiber is highly valued in various industries, particularly the textile industry. Due to its inherent benefits, wool is essential in clothing and industrial applications.

Wool's sustainability is another key factor contributing to its widespread use. Sheep naturally produce new wool every year, making it a renewable resource. This contrasts with synthetic fibers, which are often derived from petrochemicals and have a significant impact on the environment.

Wool has developed mechanisms to reduce bacterial and fungal attacks in natural conditions. The cuticle cells of wool create an uneven surface on the fiber, which makes it more difficult for microbes to adhere. The lipid layer of the epicuticle may also have antibacterial properties, as may some of the compounds that give wool its characteristic odor.

Wool fibers exhibit a unique combination of durability and elasticity, which makes them suitable for various applications, although they face certain limitations [1].

Wool is inherently soft, durable, and highly resistant to wear and tear, contributing to its longevity in different uses. Wool fibers' elasticity is remarkable because they can return to their original shape after deformation, which is beneficial for applications requiring flexibility and resilience. However, environmental factors such as humidity and UV exposure can significantly affect wool's mechanical properties.

Of all commonly used textile fibers, wool is the most resistant to burning. It is not easy to ignite; it expands slowly and extinguishes quickly. The residue is a brittle low-temperature ash that does not stick together (unlike acrylic, polyamide, and polyester fibers).

The natural properties that contribute to the low flammability of wool are:

1. high ignition temperature (750 - 800°C);
2. high limiting oxygen index (25 - 26%);
3. high nitrogen content (16%);
4. high moisture content (10 - 14%);
5. non-melting and non-flowing/splashing [17,46].

For centuries, wool has been used for warmth in clothing and bedding, but now it is also growing in popularity as a home insulation material. Wool has a natural temperature-buffering effect, which has been shown to reduce temperature changes throughout the day, saving energy when heating and cooling. Wool insulation materials are attractive to the consumer because they are easy to handle, are safe to use and come from a renewable resource [2,3,16,19].

Wool is increasingly used in technical applications where its unique properties and opportunities for specific improvements can be profitably utilized. Contrary to common belief, wool is a highly technical fiber and its presence in technical textiles must be addressed. For example, wool has traditionally been the fiber of choice in many technical applications due to its naturally low flammability characteristics.

Keratin is the most abundant fibrous structural protein in wool, hair, skin, horns, hooves and feathers of birds. Millions of tons of keratin waste are generated annually globally, mainly in the wool textile industry and in poultry slaughterhouses.

More than 5 million tons of keratin-containing by-products are produced annually, but currently these by-products have limited applications, such as use in fertilizers or biodegradable surfactants [8,9,12].

Disposal of keratin-containing materials by incineration can be problematic for the environment as they contain 3 - 4% sulfur, which can contribute to significant pollution.

Chemical hydrolysis is a method of keratin extraction involving the use of acid, base and catalyst. This process requires the application of high temperature and pressure. Various acids, including hydrochloric acid, sulfuric acid, and peracetic acid, can be used to extract keratin by an acid process [21].

Oxidative methods have long been used for keratin solubilization. Substances such as peracetic acid, hydrogen peroxide, performic acid, or potassium permanganate can partially disrupt disulfide bonds in keratin tissues and convert cystine to cysteic acid residues by oxidation.

The oxidized keratins, known as keratases, undergo chemical modifications and can be extracted and separated into different fractions (α-, β- and γ-keratases) based on their solubility at various pH levels [21].

CHAPTER IV. STATISTICAL SURVEY ON THE ECONOMIC IMPORTANCE OF SHEEP

IV.1. Materials and methods

Data collected from the statistical program on the Knoema platform (https://knoema.com/) were examined to assess the environmental impact of sheep wool waste.

This statistical study, which relies on a trusted and comprehensive data source, has followed sheep and wool production fluctuation at global, European, and national levels for eleven years.

Following data collection, we conducted a rigorous and comprehensive analysis to evaluate the environmental implications of our findings. This thorough approach, which leaves no stone unturned, ensures the validity and reliability of our results.

Our data analysis and graph preparation were conducted with the utmost care and precision using Microsoft Office Excel 2021. This robust approach has allowed us to present our findings accurately and comprehensively.

IV.2. Results and discussion

The following figures present the statistical study results, which focused on tracking the fluctuations in sheep and sheep wool production at global, European, and national levels for eleven years.

World sheep production

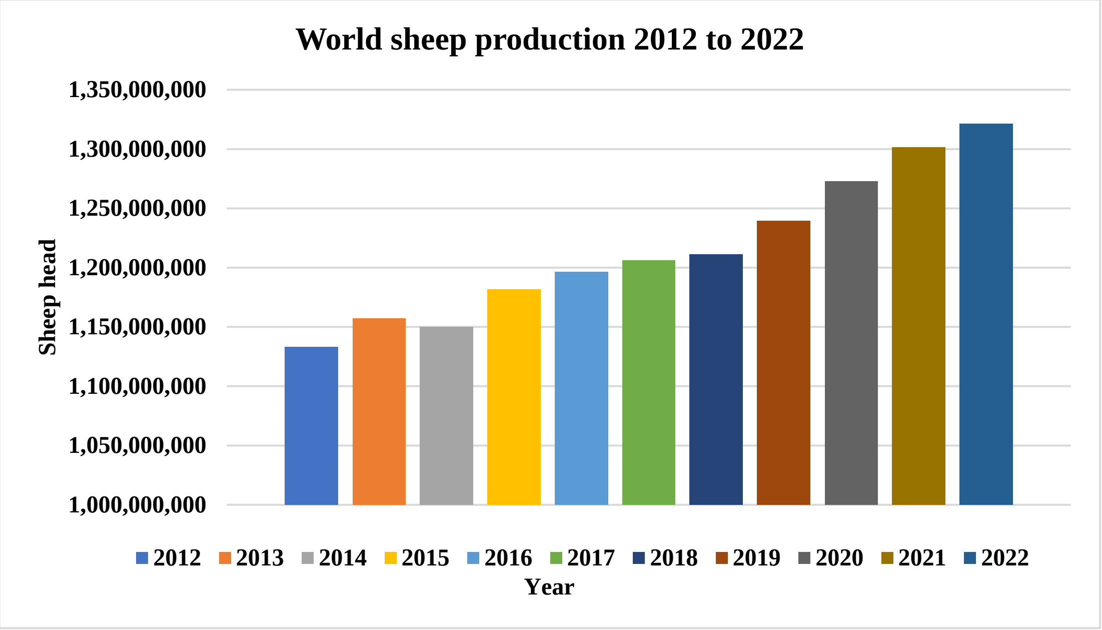

Figure 1. World sheep production 2012-2022

World sheep production in 2022

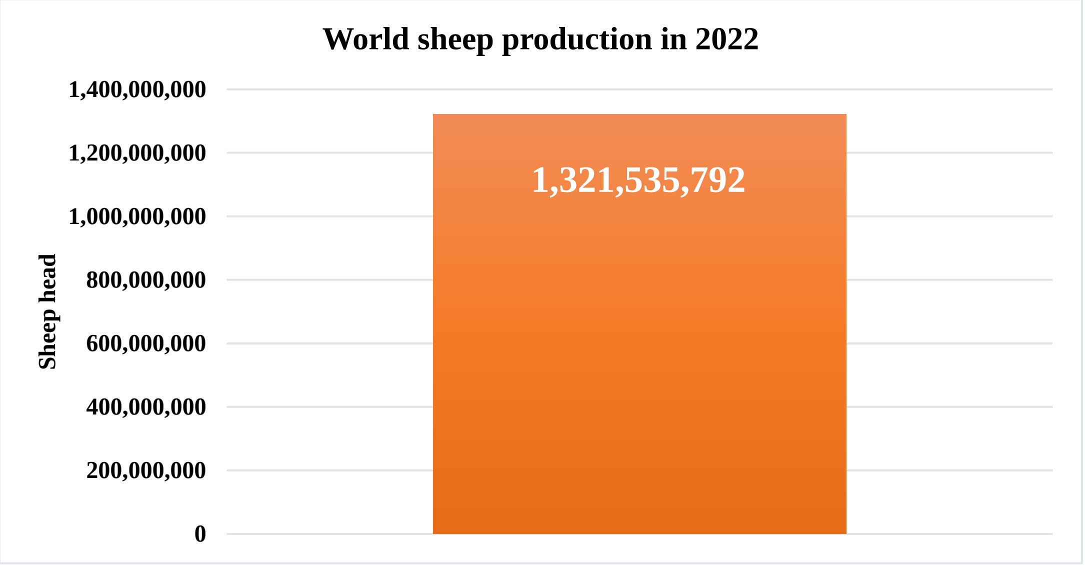

Figure 2. World sheep production in 2022

Sheep production in Europe

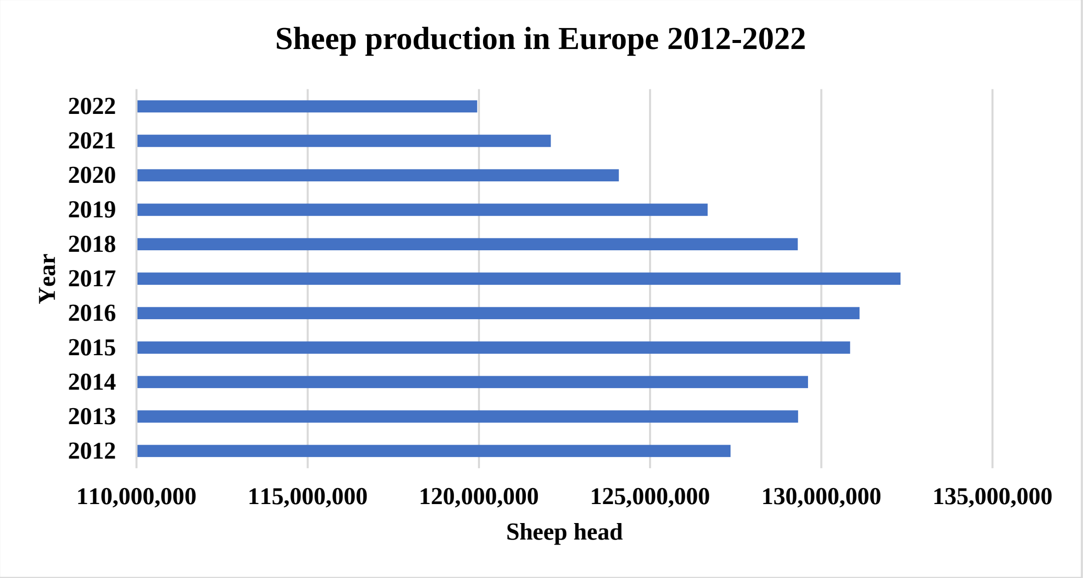

Figure 3. Sheep production in Europe from 2012 to 2022

Sheep production in different regions of Europe

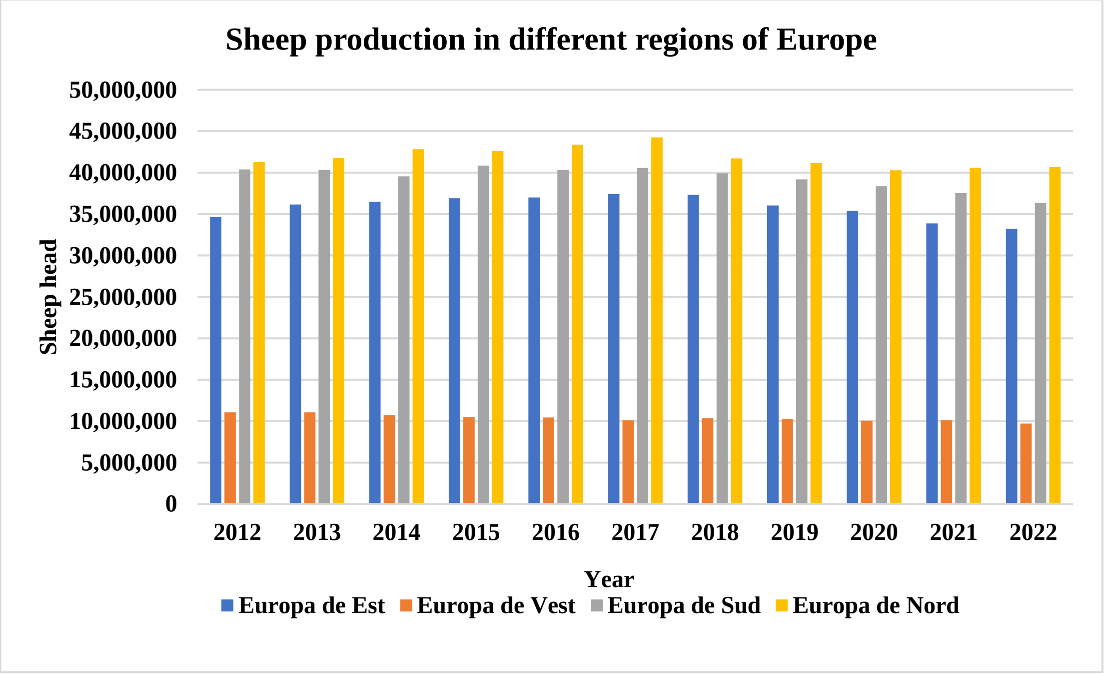

Figure 4. Sheep production in European regions (2012-2022)

Sheep production in EU member countries from 2012 to 2022

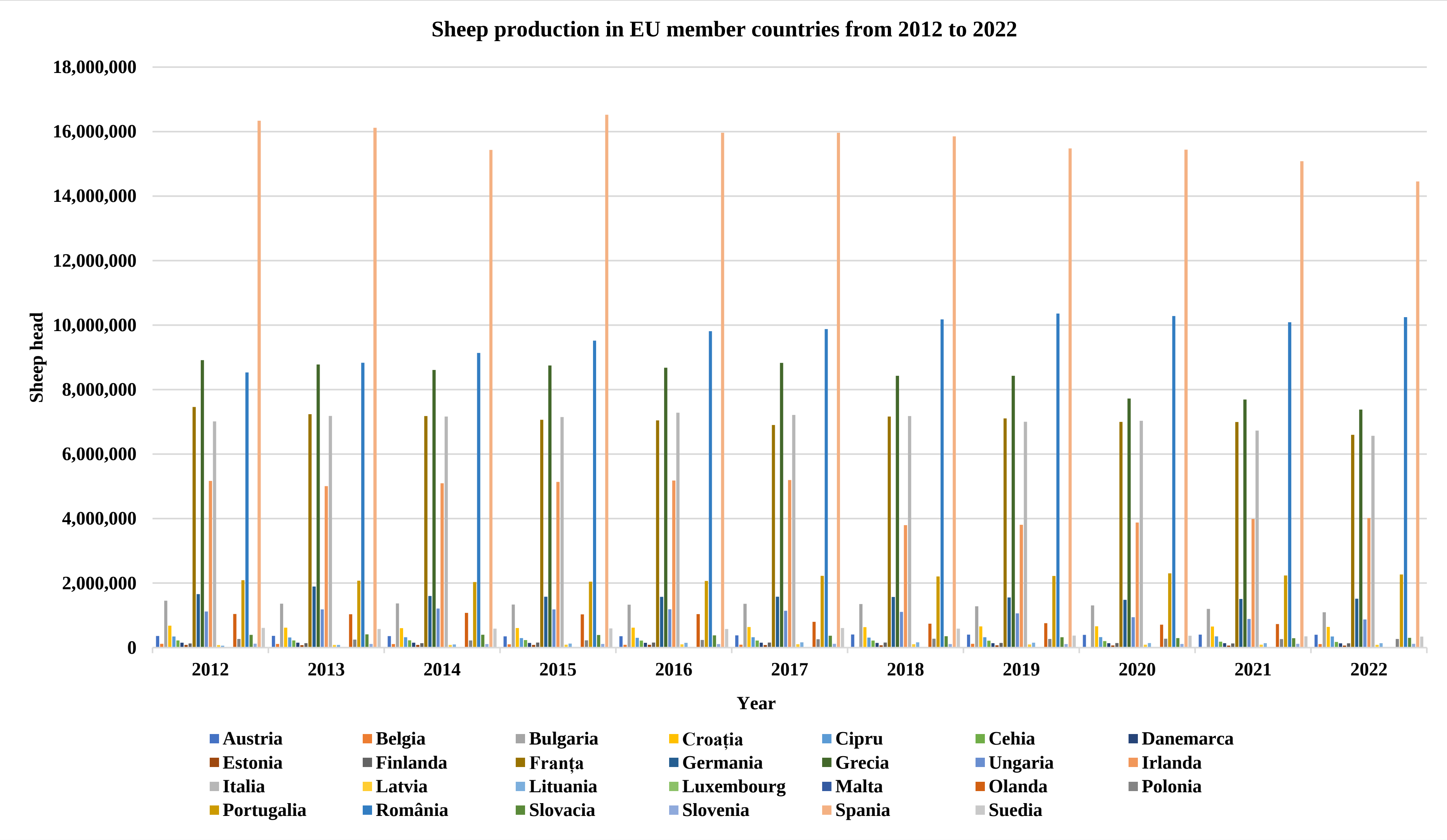

Figure 5. Sheep production in EU member countries from 2012 to 2022

Sheep production in EU member countries in 2022

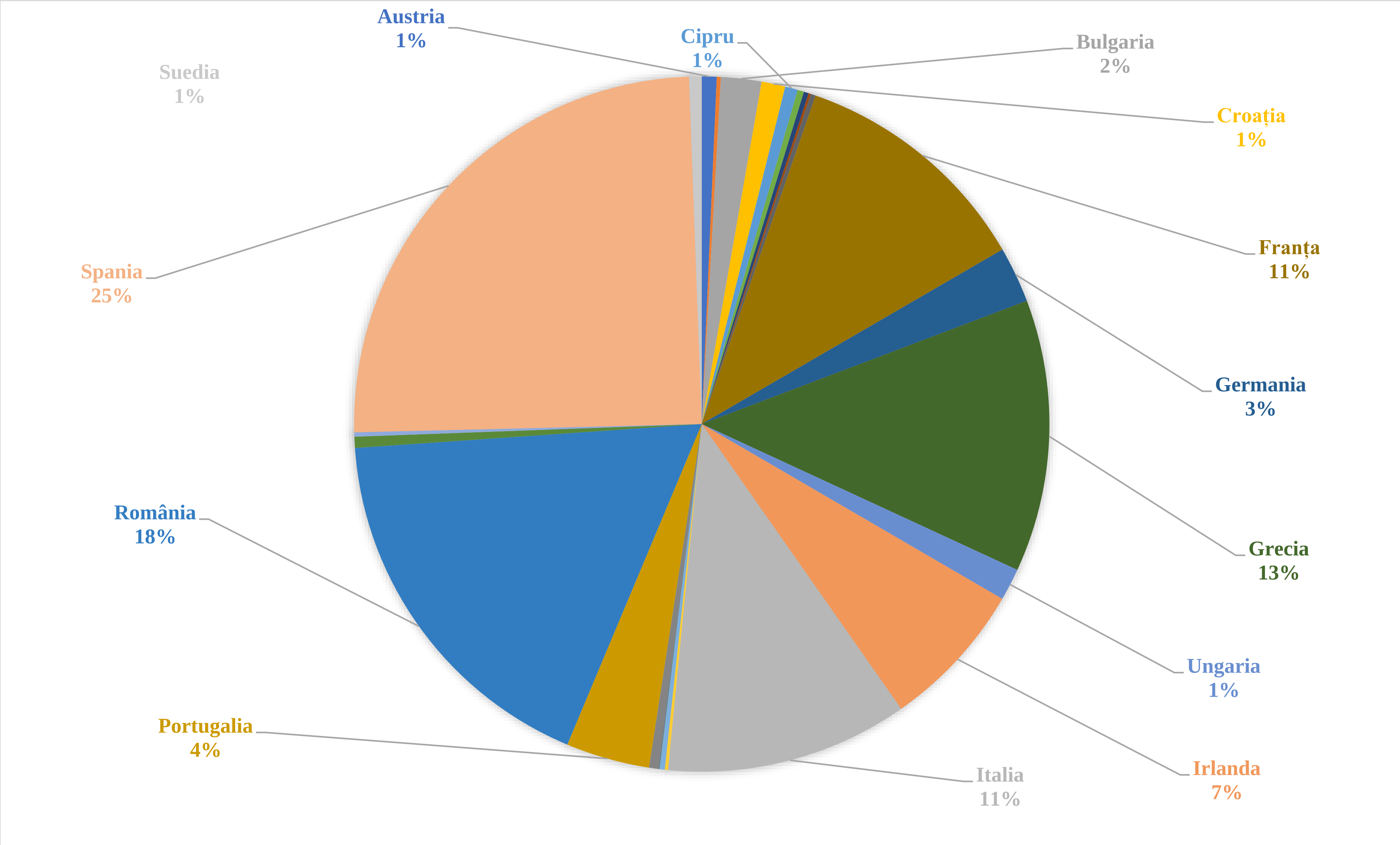

Figure 6. Sheep production in EU countries in 2022

Sheep production in Romania

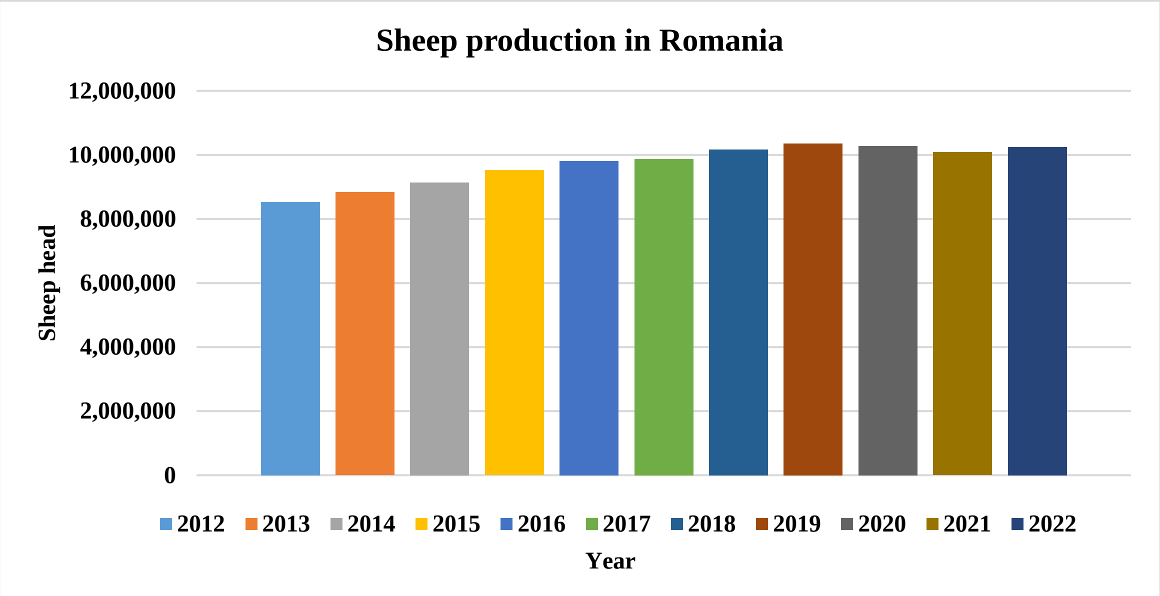

Figure 7. Sheep production in Romania from 2012 to 2022

World wool production

Figure 8. World wool production from 2012 to 2022

Wool production in Europe

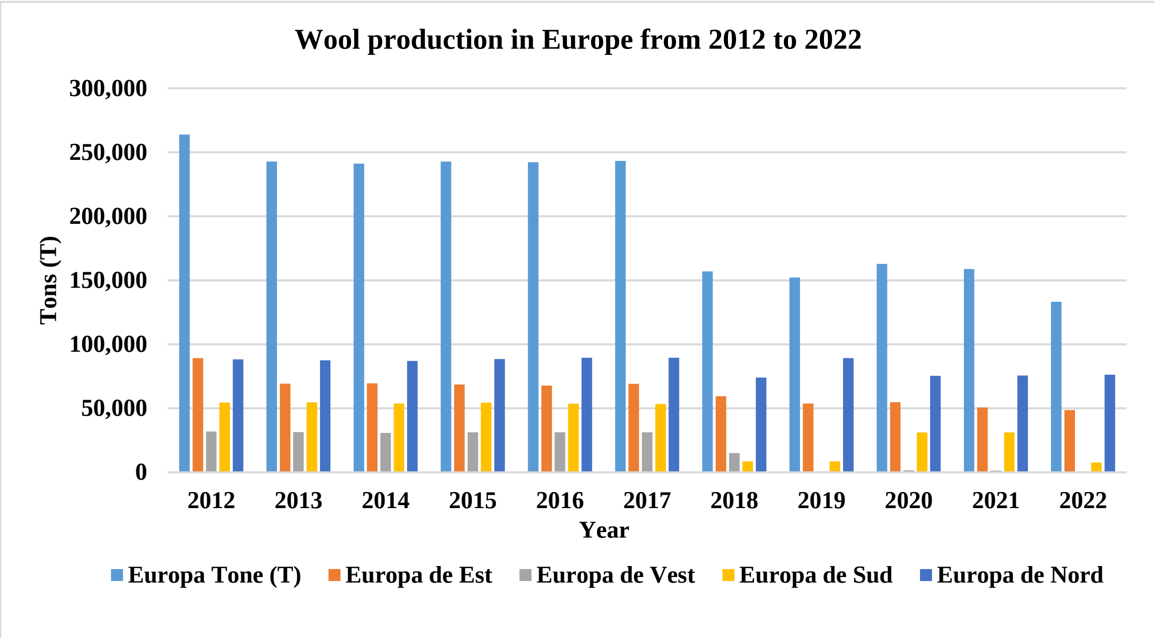

Figure 9. European wool production 2012-2022

Wool production in Romania

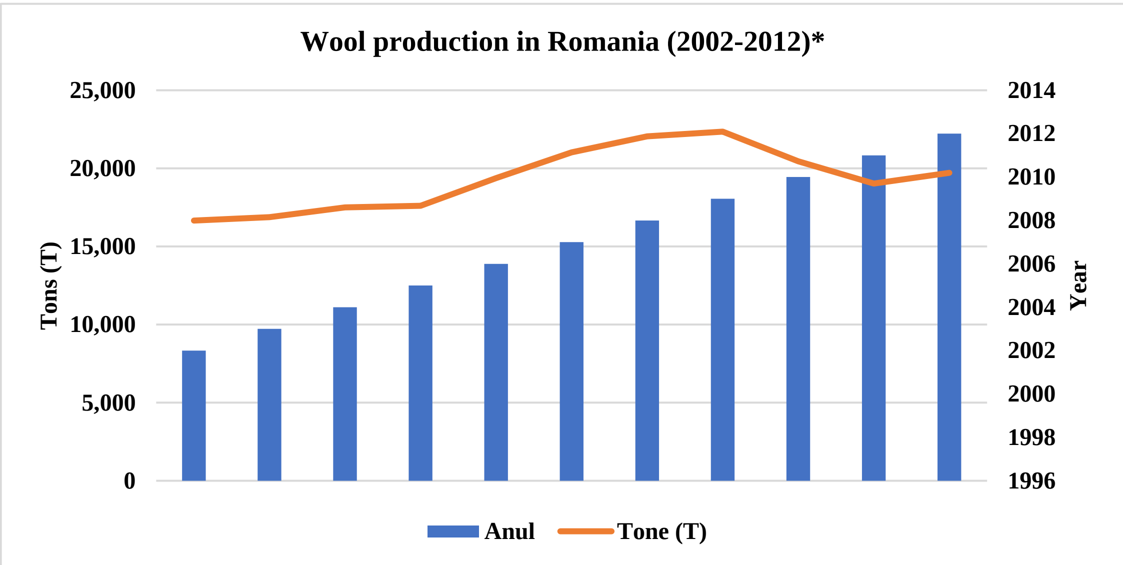

Figure 10. Wool production in Romania (2002-2012)*

Sheep wool production recorded as waste

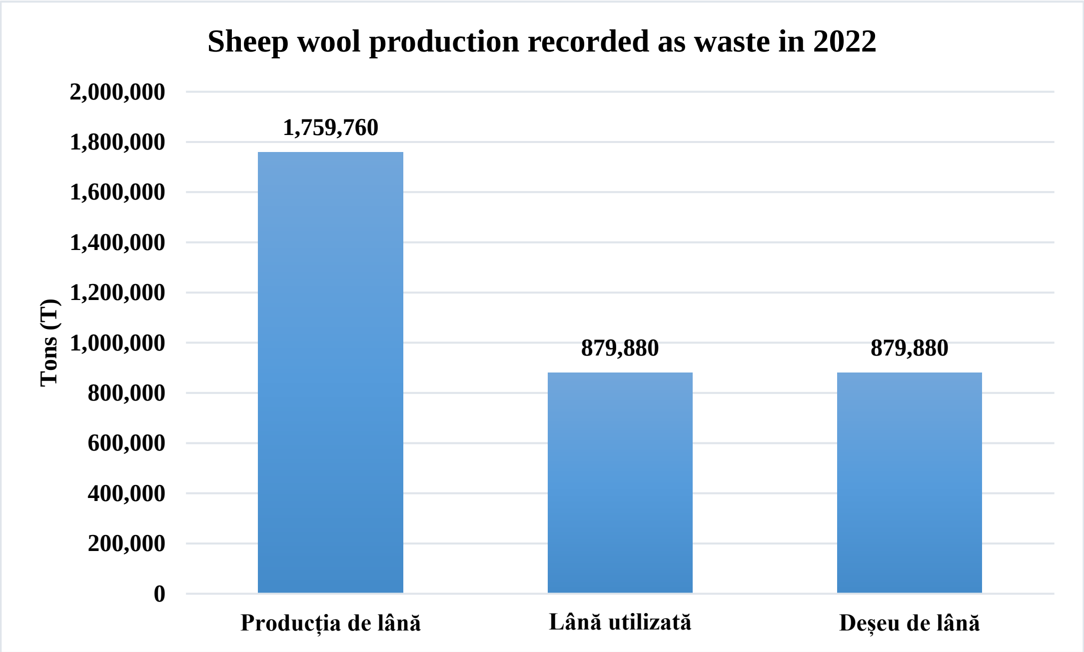

Figure 11. Production of sheep wool recorded as waste

IV.3. Conclusions and recommendations

Sheep farming is vital to the economies of many countries worldwide and is an essential key to rural development, particularly in mountainous and arid regions.

The eleven-year statistical study reveals that the annual Global, European, and Romanian wool production yields a substantial volume of wool fiber. While the textile industry utilizes a portion of this, the remaining wool, a significant environmental waste [17], poses a pressing concern.

This has become a pressing environmental concern as sheep wool has become a significant pollutant, necessitating immediate action.

There has been an increasing emphasis on the sustainable use of natural resources, which has led to the re-evaluation of wool as a valuable renewable resource. This underscores the importance of your role in the textile industry, as significant research has focused on finding ways to utilize waste [23].

CHAPTER V. STUDY ON THE EFFICIENCY OF SHEEP WOOL FIBERS TO RETAIN HEAVY METALS IN POLLUTED WATERS

V.1. Proposed objectives

In this experimental study, we observed the remarkable efficiency of wool filters in retaining heavy metals from polluting waters in two stages. This efficiency, as we will demonstrate, holds significant potential for addressing water pollution issues.

The first stage involved observing the retention efficiency of heavy metals from contaminated waters using a fixed-phase approach. Therefore, different solutions with specific concentrations of heavy metals were prepared, and the wool filter was immersed in these solutions for different durations.

The second stage of this experimental research study involved observing heavy metal retention in polluted waters from a mobile phase perspective. Specifically, the effectiveness of the wool filter was examined by testing it at different flow rates.

This work will present the results of the two experimental stages, each meticulously designed and executed to provide a comprehensive understanding of wool filters' efficiency in retaining heavy metals in polluted waters.

V.2. Materials and Methods

For the experimental part of this work, sheep wool from sheep shearing was used. It was obtained in June 2023 from a local sheep farm in Arad, Romania. The chemicals used were cadmium sulfate ($CdSO_4$), magnesium ($MgSO_4$), copper ($CuSO_4$), and distilled water.

The instruments used were a Masterflex Easy-Load II pump (Avantor, Radnor, Radnor, Pennsylvania, USA) and a Specord 200 spectrophotometer (Analytik Jena, Jena, Germany).

It is crucial to understand that sheep wool waste from wool shearing, which is the focus of this research study, has yet to be recovered or utilized. Instead, it is discarded and becomes an environmental pollutant.

This research aims to highlight the importance of its findings by demonstrating the potential environmental impact of this waste and the urgent need for effective remediation methods.

The sheep shearing process was carried out by hand, producing good-quality wool. On the shearing day, the wool was collected in plastic bags and then transported to the laboratory for analysis.

FIXED PHASE

Preparation of samples for analysis

For the first step, the fixed phase, 2g of clean, dry sheep wool was prepared and weighed. Solutions of $CdSO_4$, $CuSO_4$, and $MgSO_4$ 0.1 M/mL were prepared in a total volume of 100 mL.

The wool sample was immersed in the solution for 30, 60, and 90 min. After each of the above-mentioned time intervals, 10 mL of the sample was extracted and prepared for spectrophotometric analysis. The absorbance was measured at the wavelength according to the analyzed sample: cadmium—193 nm, copper—807 nm, and magnesium—190 nm [4,5,15,18].

Optimization of wool filters

The changes were the type and amount of wool used and the preparation of a single heavy metal solution; the rest of the working method remained the same. The first step in sample preparation was weighing 6g of clean, dry wool. The solution's concentration, amount, and contact time have remained unchanged.

The wool used in this experiment is from the Cărăbașă breed of sheep, an autochthonous Romanian breed known for its exceptional adaptability to various environmental conditions. This ensures the reliability and consistency of the wool used in our experiment.

In a total volume of 100 mL, 0.1 M/mL, the wool sample was immersed in the cadmium solution for 30, 60, and 90 minutes. After each mentioned time interval, 10 mL of the sample was extracted and prepared for spectrophotometric analysis.

Since temperature is a significant factor, the experiment was conducted in a controlled environment at room temperature, specifically ±22°C.

MOBILE PHASE

Preparation of samples for analysis

Sheep wool filters showed great promise in efficiently trapping heavy metals from contaminated water.

The results are encouraging, indicating that wool has the potential to serve as a cost-effective and durable material for water filtration.

This potential offers hope for developing sustainable and efficient water treatment methods.

Table 1. The flow rates depending on the speed used

The flow rates depending on the speed used	
Speed 1	2.5 mL/min
Speed 2	7.2 mL/min
Speed 3	11.2 mL/min
Speed 4	15.2 mL/min

The flow rates at which contaminated water passes through sheep wool filters may influence their efficiency in retaining heavy metals.

3g of clean, dry sheep wool was prepared and weighed. A 100 mL volume of $CdSO_4$ 0.1 M/mL solution was prepared.

The solution was recirculated three times, and after each recirculation, 5 mL of the sample was extracted and prepared for spectrophotometric analysis.

V.3. Results and discussion

Fixed phase results

To establish a balance between sheep wool waste and the environment, different scientific studies have tried to find cheap and effective solutions to combat this problem over the years. Thus, numerous research studies have led to a promising breakthrough in the recovery of sheep wool waste, namely the design of filters and the observation of their efficiency in retaining contaminants and heavy metals from industrial wastewater and pollutants.

Efficiency of sheep wool for the retention of cadmium ions

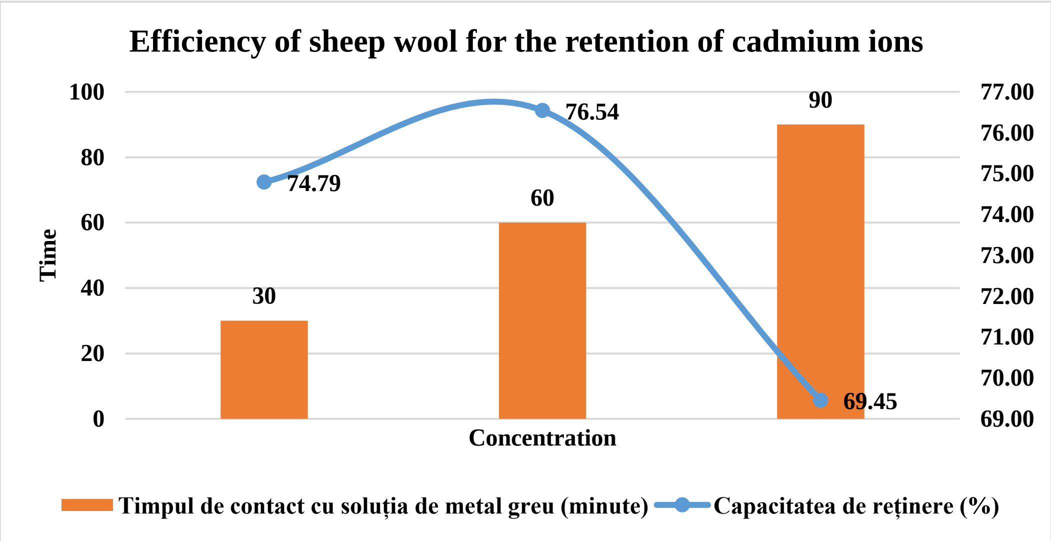

Figure 12. Efficiency of sheep wool in retaining cadmium ions

Efficiency of sheep wool for retaining copper ions

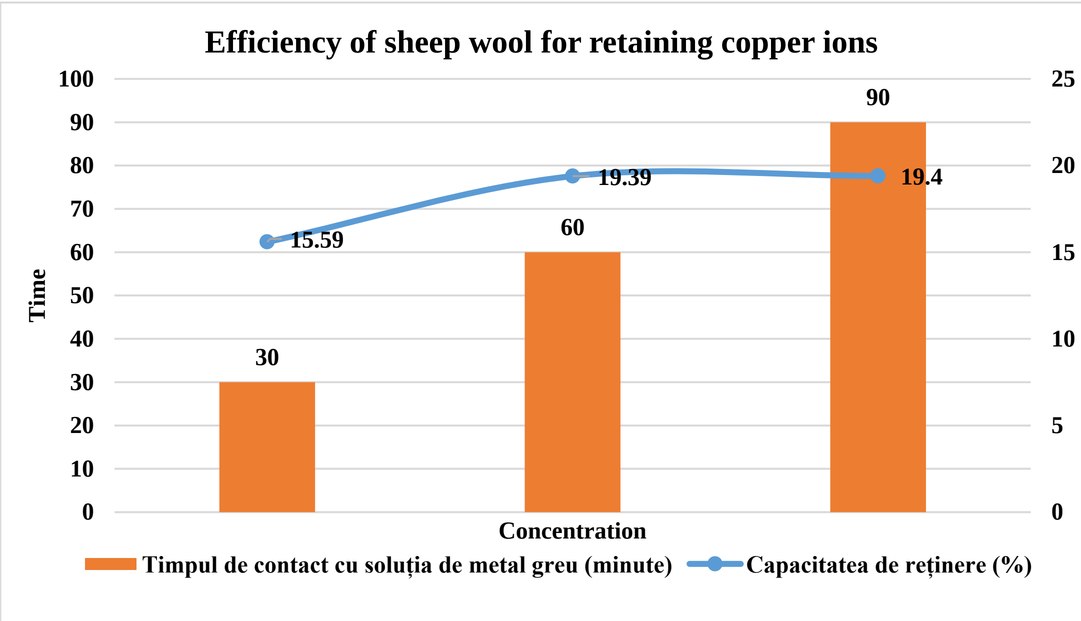

Figure 13. Efficiency of sheep wool in retaining copper ions

Efficiency of sheep wool for retaining magnesium ions

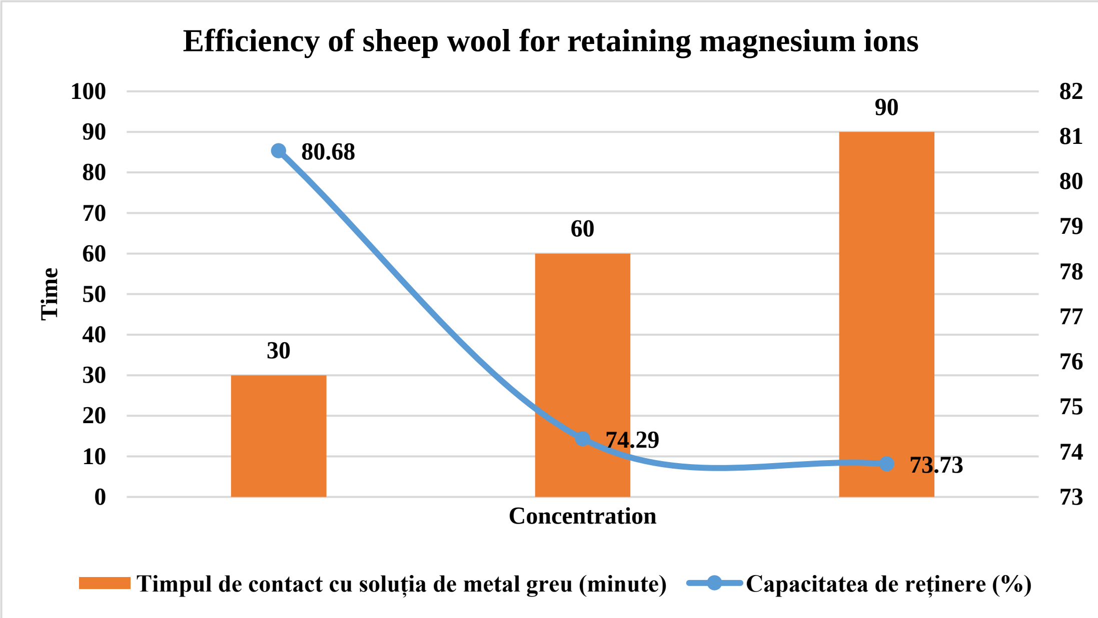

Figure 14. Efficiency of sheep wool for retaining magnesium ions

Results of the wool filter optimization process

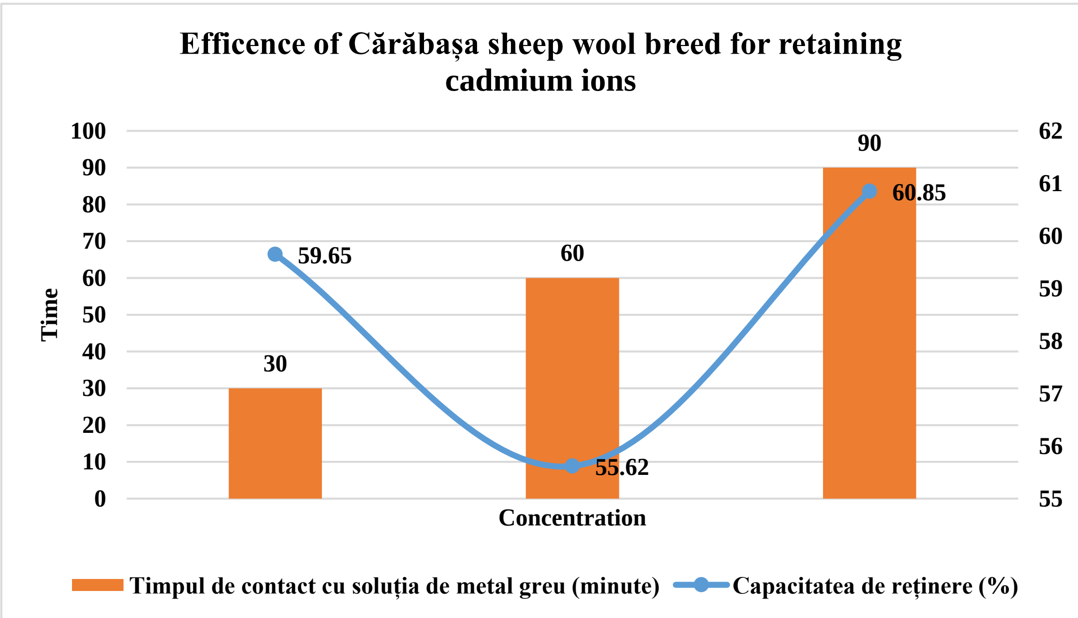

Figure 15. Efficiency of sheep wool for the retention of cadmium ions

Mobile phase results

The efficiency of the wool filter was observed as a function of flow rate by recirculating the $CdSO_4$ solution three times.

Speed 1

The applied flow rate was 2.5 mL/min.

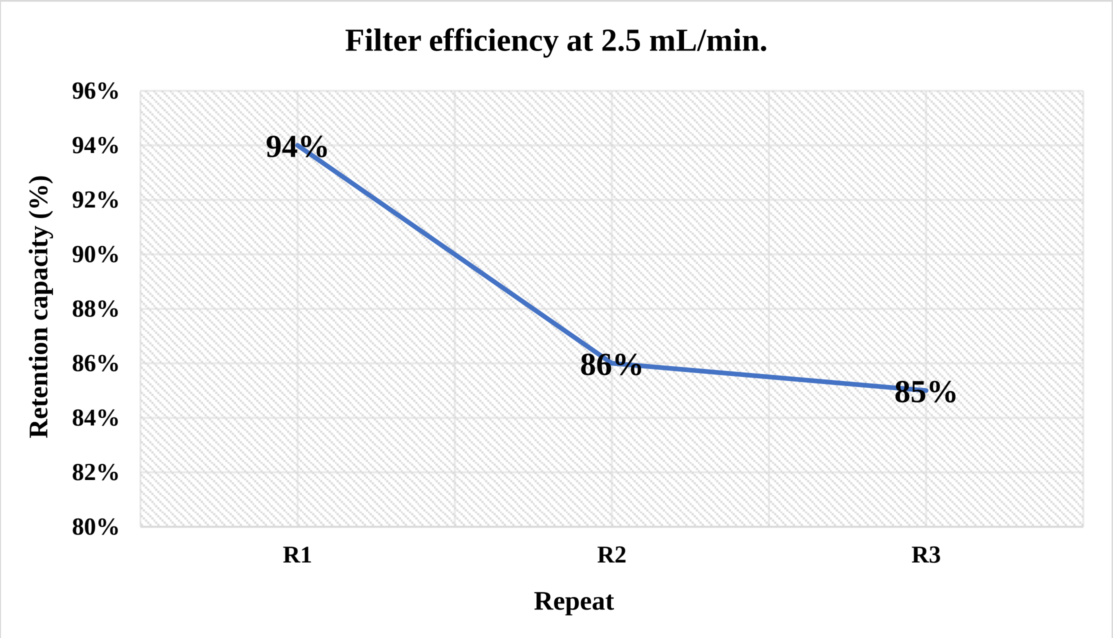

Figure 16. Efficiency of recirculation filter applying a flow rate of 2.5 mL/min.

Speed 2

The applied flow rate was 7.2 mL/min.

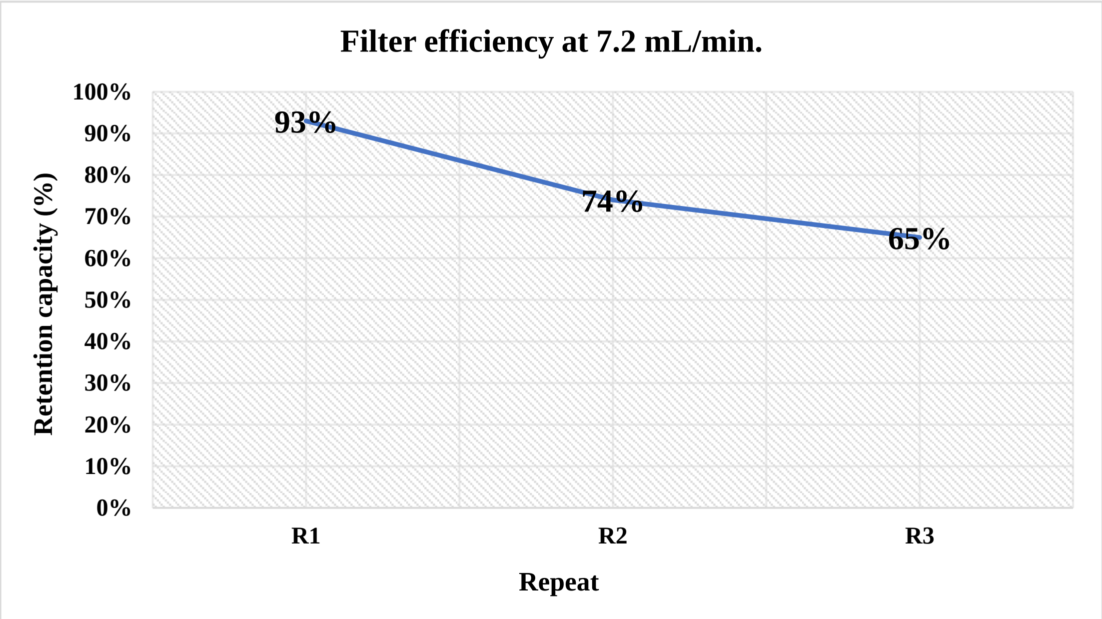

Figure 17. Efficiency of recirculation filter applying a flow rate of 7.2 mL/min.

Speed 3

The applied flow rate was 11.2 mL/min.

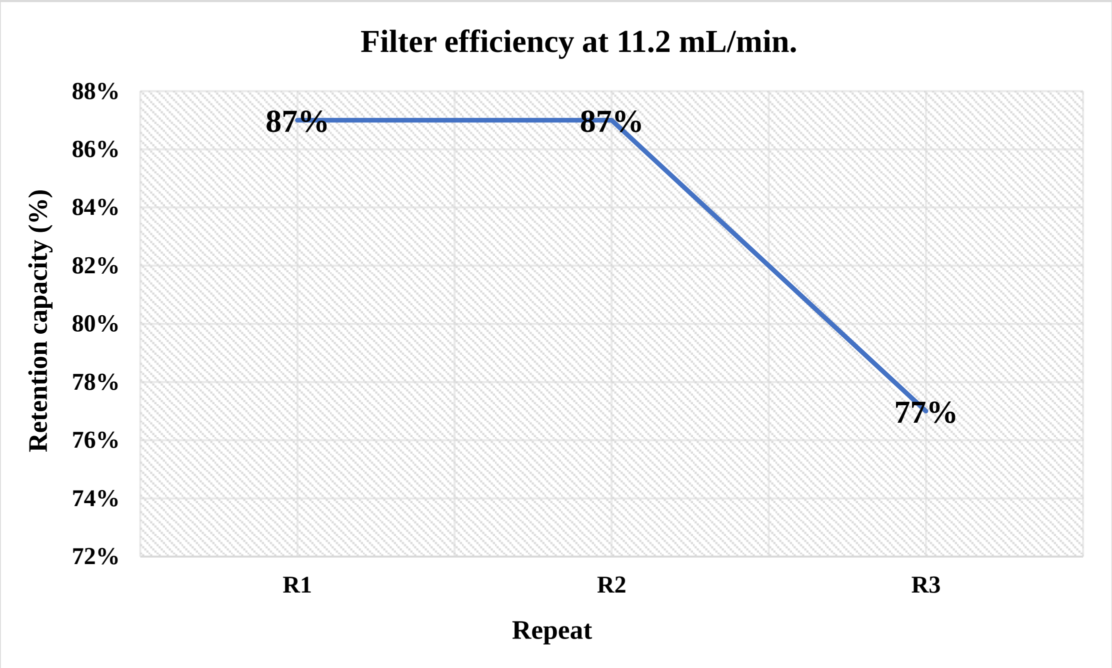

Figure 18. Efficiency of recirculation filter applying a flow rate of 11.2 mL/min.

Speed 4

The applied flow rate was 15.2 mL/min.

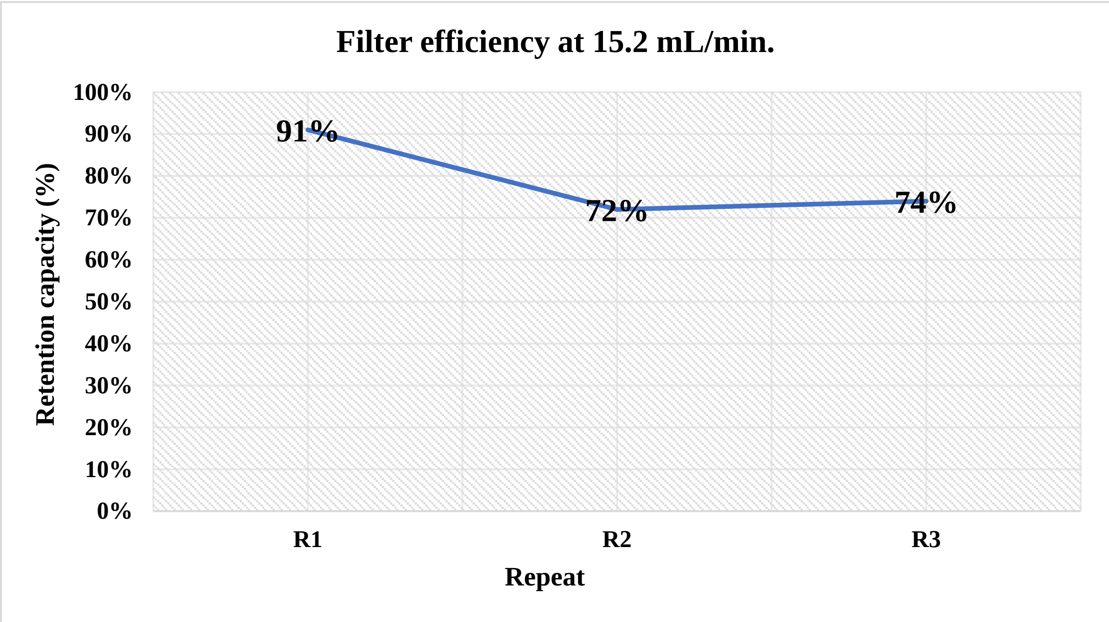

Figure 19. Efficiency of recirculation filter applying a flow rate of 15.2 mL/min.

V.4. Conclusions and recommendations

Based on the results, sheep wool filters have shown great promise in effectively retaining heavy metals from industrial wastewater. Sheep wool has remarkable attributes that make it a very effective filter material and a remarkable capacity to absorb heavy metals.

The results obtained from the first fixed-phase stage of the research study demonstrate the effectiveness of sheep wool-based filters in retaining the heavy metals studied. In addition, also in the fixed-phase stage of the research study, wool filters show a significant improvement in their ability to retain cadmium sulfate.

The optimum flow rate may vary depending on the specific conditions and contaminants; a flow rate of approximately 7.2 mL/min, combined with sufficient contact time, is effective for heavy metal retention in sheep wool filters.

In conclusion, the analysis and the promising results obtained from both steps we followed in this research study show that sheep wool's complex properties make it a very versatile and practical material for treating environmental problems.

GENERAL CONCLUSIONS

Using sheep wool waste represents a significant step forward in sustainable resource management, effectively addressing economic and environmental obstacles. Sheep wool, commonly regarded as a lower-value by-product of the wool and meat sectors, has been underutilized and often discarded as waste. However, new recovery methods demonstrate its potential in several industries [11,22].

Sheep wool waste has shown great potential in environmental remediation, particularly water filtration, to remove heavy metals and other contaminants. This method is very promising for solving environmental problems. Due to its outstanding physical and chemical properties, sheep wool is very effective in adsorbing heavy metals from polluted water.

In addition to environmental applications, sheep wool waste is used in various fields such as agriculture, construction, and textiles. Wool waste has proved to be a valuable resource in agriculture, serving as a natural manure and soil fertilizer.

Its application can greatly improve soil health and promote optimal plant growth. Wool waste is used in the construction industry as an insulating material due to its exceptional thermal and acoustic properties. Furthermore, advances in textile recycling facilitate the transformation of wool waste into new textile products, promoting a circular economy [11].

Utilizing sheep wool waste is a solution to environmental and economic obstacles that need to be increasingly addressed. By transforming waste into valuable resources, the potential exists to minimize environmental pollution, promote economic development, and progress toward a sustainable and resilient future.

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