Scientific Papers. Series E. Land Reclamation, Earth Observation & Surveying, Environmental Engineering. Vol. XIII, 2024
Print ISSN 2285-6064, CD-ROM ISSN 2285-6072, Online ISSN 2393-5138, ISSN-L 2285-6064

RESEARCHES FOR THE PHYSICO-CHEMICAL CHARACTERIZATION OF SOME GRANULAR FERTILIZERS OBTAINED FROM NATIVE WOOL WASTE, IN ORDER TO USE THEM IN AGRICULTURE

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Abstract

Wool waste production cannot be avoided, so reuse and recycling are the best solutions for managing this keratinous waste. At present, in Romania, wool in different forms - raw or washed wool, pelleted wool or wool hydrolysate - is used in agriculture on a relatively small scale, both as a fertilizer and to improve certain physical characteristics of the soil. A good knowledge of the physico-chemical properties of wool-based fertilizer granules will also contribute to increasing the use of this resource. In this research, the characteristics of some wool granules produced in Romania were studied, such as bulk density, granule moisture, water absorption, and the effect of alkaline hydrolysate obtained from them on soil-beneficial bacteria of the Bacillus genus and Rhizobium genus. Among the results obtained, it should be mentioned that the water absorption capacity of the granules is around 200%. Also, the research proved that the alkaline hydrolysate obtained from the wool pellets studied does not show inhibitory effect on the Bacillus and Rhizobium species tested.

Key words*: characteristics, granular fertilizer, hydrolysate, wool.*

INTRODUCTION

Total world wool production and wool prices have steadily declined over the last decades. According to recent figures, world wool production is 1950 million kg (IWTO, 2021) down 0.6% from 2015, and represents 1.73% of total fibre production (113 million tons) (statista.com).

According to a 2019 report, it is estimated that approximately 10-15% of wool is wasted during sorting and cleaning, and an additional 12-15% is lost during spinning and weaving (Kadam et al., 2014; Sharma et al., 2019).

Disposal of wool waste by incineration and landfilling causes air and soil pollution when the amount of wool is not carefully controlled. (Kumawat, 2018; Marchelli et al., 2021).

In order to overcome these pollution problems and to use this large amount of waste as a resource, it has been proposed over the years to use wool waste for numerous applications such as insulation (Cai et al., 2021), building materials (Buratti et al., 2020), adsorbents (El-Geundi, 1997), composite materials (Remadevi et al., 2020), mulches or fertilizers for agriculture (Ordiales et al., 2016).

The advantage of wool is given by its amidic structure that can bring benefits to the soil by retaining water and mineralizing nitrogen, wool is also biodegradable within six months under ideal conditions (Haque & Naebe, 2020). The use of wool for plant growth is sustainable and can benefit the soil if used rationally, which can reduce the amount of mineral fertilizer to a minimum, if not replacing it altogether (Wiedemann et al., 2020).

Furthermore, evaluation of cleaned wool residues obtained from textile mills in terms of their effects on soil properties demonstrated that their use improves bulk density, water holding capacity and soil aggregation (Abdallah et al., 2019).

There is also an increased interest in the development of more complex wool-based fertiliser systems that incorporate other components into their structure. One such example is the application of a biostimulant

containing humic acid, lactic acid and *Bacillus subtilis* in the rhizosphere in parallel with the use of unbleached sheep's wool plates as substrate for hydroponic cucumber production (Böhme et al., 2005). Recent studies have also investigated the potential use of arbuscular mycorrhizal fungi in combination with wool pellets for agricultural crops (Larsson, 2022). However, there is no information on the potential of using wool as a carrier for immobilisation of different types of bacteria with important roles in the soil (e.g. nitrifying bacteria, lactic acid bacteria) or on the combined use of wool in various forms in mixture with lyophilisates of these bacterial species for fertilisation, amendment or soil conditioning. Such a research direction is in line with European Circular Economy policies, and from this point of view, coarse wool and other related waste should be treated as a secondary raw material and used as a valuable source of nutrients and organic matter.

In this context, the paper present the research regarding the characteristics of some wool granular fertilizer produced in Romania and the effect of alkaline hydrolysate obtained from them on soil-beneficial bacteria of the *Bacillus* genus and *Rhizobium* genus.

MATERIALS AND METHODS

The wool pellets used for the experiments were purchased directly from the producer, SC Miorita, Ucea de Jos, Brasov. According to the information from the manufacturer the pellets were made from raw, milled wool, with the fibre size being about 6 mm, after which it was processed with a pelleting press. Two types of pellets were made with different diameters, 6 mm (S6) and 9 mm (S9) (Figure 1).

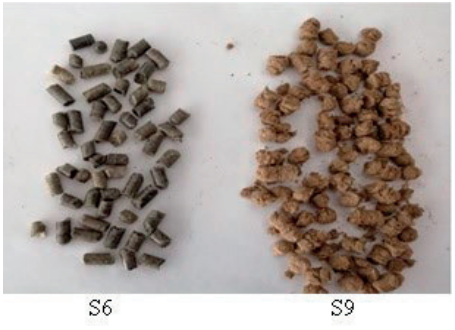

Figure 1. Wool pellets used for experiments

Some of the main quality indicators of wool pellets are presented in Table 1.

Table 1. Wool pellets quality indicators (analysis bulletin Raiffeisen Laborservice)

No.	Quality Indicators	U. M.	Determined values
1	Dry matter	%	89.30
2	pH measured at 20.6 ^{0}C	pH units	9.56
3	Density	g/l	0.33
4	Nitrogen	% D.M	12.14
5	P_2O_5	% D.M.	0.18
6	K_2O	% D.M	5.08
7	Sulphur	% D.M	2.11
8	CaO	% D.M	0.46
9	MgO	% D.M	0.19
10	Organic matter	g/kg D.M.	847.4
11	Humus-C	g/kg D.M.	460.4
12	Copper	mg/kg D.M	8.1
13	Iron	mg/kg D.M	2296
14	C:N ratio		3.8 :1

To determine the water content, we used samples of about 3 g of granules, which were dried at 70^{o}C, with a thermobalance type AXIS-100 with a weighing accuracy of 0.01%, (Figure 2). At every 30 seconds the masses were recorded until a constant mass has been obtained. Moisture content is given by the difference between the initial and final mass of the samples.

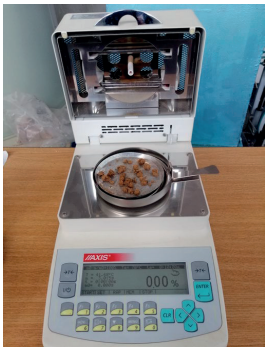

Figure 2. Drying wool pellets with thermobalance Axis-100

To determine the water absorption capacity of the wool pellets, 5 g samples were weighed over which distilled water was added in 10 ml fractions, with a 5-minute break between fractions, until excess water remained. The excess water was removed with absorbent paper and reweighed the saturated sample.

The water absorption relative to mass was calculated with the formula (1):

$a_m=(m_{sa}-m_{us})*100/m_{us}$ (1)

where:

- m_{us} – pellets dry mass, g;
- m_{sa} – pellets saturated mass, g;
- a_m – water absorbtion relative to mass, %.

To determine the bulk density of the wool pellets we used a calibrated vessel of volume V = 0.20 dm^3 with mass m_{vas} = 107.75 g. To obtain the mass m_1 we poured the granules into the vessel from 5 cm height, then we buffered it for around 30 times by a wooden table and weighed again. The measurements were repeated by 5 times to determine the average value of the m_1. Compact bulk density was calculated using the formula (2):

$\rho = (m_1-m_v) / V$ (2)

where:

- m_v –vessel mass, g;
- V –vessel volume, dm^3;
- m_1 – mass of the vessel with pellets, g.

In order to increase the fertilizer and soil amendment qualities of wool pellets by the addition of beneficial plant and soil-borne telluric microorganisms, the effect of hydrolysates obtained from the pellets on beneficial bacteria such as *Bacillus genus* and *Rhizobium genus* was studied.

For this the alkaline hydrolysis of wool pellets was carried out and slightly modified according to the previous method described by Berechet and colleagues, which has been declared as suitable for wool by-products and wool itself (Berechet et al., 2018).

For this research bacterial cells of *Bacillus subtilis*, *Bacillus subtilis USAB*, *Bacillus subtilis B 52*, *Bacillus subtilis B36*, and *Bacillus licheniformis 1*, as well as *Rhizobium Gz 13*, *Rhizobium meliloti*, *Rhizobium trifolii*, *Rhizobium meliloti USAB* and *Rhizobium lupini LP 83 FM*, were cultured under aerobic environmental conditions at USV Timisoara.

To assess whether the resulting hydrolysate samples exhibit potential bacterial inhibitory properties, the disc diffusion assay was used for verification, as previously reported (Balta et al., 2021). Briefly, 100 µl of liquid bacterial cultures of *B. subtilis*, *B. subtilis USAB, B. subtilis B 52, B. subtilis B36 and B. licheniformis 1* were inoculated onto Petri dishes with nutrient agar and spread with a Drigalsky spatula. Similarly, 100 µl of *R. GZ 13, R. meliloti, R. trifolii, R. meliloti USAB and R. lupini LP 83 FM* were placed and spread on yeast mannitol agar plates. Next, 6 mm paper discs were placed in each plate and hydrolysate samples at 20 µl were absorbed onto the paper discs at the specified hydrolysate concentration. Plates were left at room temperature for 10 min to allow better absorption of hydrolysates into the blank discs and were incubated inversely at 37°C and 28°C for the appropriate time. Antimicrobial activity of each hydrolysate was measured using the caliper as the diameter of the inhibition zone around the disc.

RESULTS AND DISCUSSIONS

Figure 3 shows graphically the results obtained from the wool pellet drying process. From the analysis of the measured data it was observed that the S6 pellets had a moisture content of 9.6% and the total drying time was 810 s, while for sample S9 the moisture content was 9% and the total drying time was 1020 s. The increase in drying time is due to the larger diameter of the pellets in sample S9.

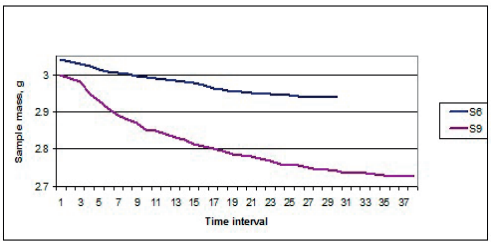

Figure 3. Drying curves at 70°C

The values obtained for the bulk density of the wool pellet's samples are presented in Table 2.

Table 2. Bulk density of wool pellets samples

	S6		S9	
	m_1, g	ρ, kg/m^3	m_1, g	ρ, kg/m^3
V_{vas}=0.20 dm^3	231.89	620.05	161.02	270.30
	230.86		162.61	
	231.72		161.63	
m_v =107.75 g	231.95		161.45	
	232.38		162.34	

Sample S9 has a much lower bulk density compared to sample S6 - which can be justified by the appearance of these pellets - much more aerated, probably because the pellet equipment

has been equipped with a sieve with larger orifices (9 mm), but the other working parameters (pressure) remained unchanged as for S6.

Analyzing the data presented in Table 3, obtained from the calculation of water absorbed in relation to mass for the two types of wool pellets, we observe that the two samples show a close water absorption capacity, sample S9 absorbing only 9% more than S6.

Also, during the experiments, it was observed that sample S9 absorbs water much faster - the first 10 ml fraction was absorbed almost instantaneously unlike sample S6 where after 5 minutes water is still observed in the vessel (Figure 4).

Table 3. Water absorbtion capacity of the wool pellets

Sample	Water added g	Dry pellets m_{us}, g	Saturated pellets m_{sa}, g	Water absorbed a_m, %
S6	20.02	4.98	15.82	218
S9	19.92	4.99	16.32	227

Figure 4. Water absorption - 5 min. after addition of 10 ml water

When the second 10 ml fraction of water was added, the S9 sample completely disintegrated in the first minute while for the S6 sample after 5 minutes pieces of pellets were still observed (Figure 5). The delay in water absorption is due to the compaction of the pellets - S6 have a much denser structure than S9.

Since the pellets are desired to decompose and release nutrients in a longer time it is obvious that the structure of the S6 sample is more advantageous.

Figure 5. Water saturated samples

After adjusting the pH of the hydrolysate obtained from the pellets to a value as close as possible to 7 (similar to the bacterial growth medium) these conditions were maintained for the next 5 days in order to determine the possible antibacterial effects of the alkaline hydrolysates prepared by the disc diffusion method. The results showed that none of the hydrolysates showed inhibitory effects on the *Bacillus* and *Rhizobium* strains tested. As can be seen in Figure 6 and 7, in all cases the bacteria grew on the agar media up to the discs impregnated with keratin hydrolysates and no zone of inhibition was observed.

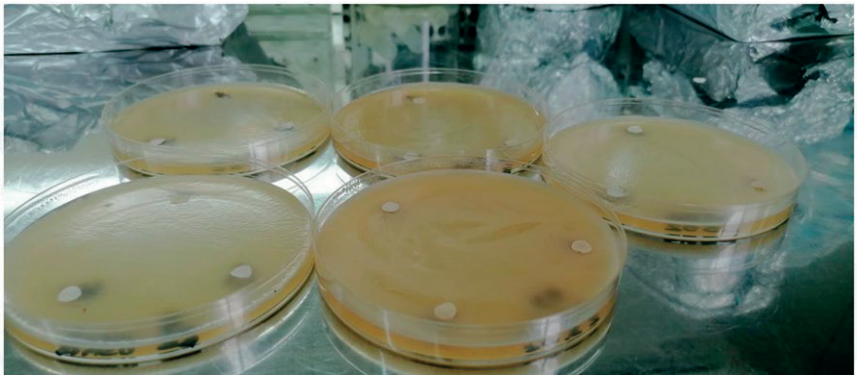

Figure 6. Plates with bacteria of the genus *Bacillus* grown on simple agar with discs impregnated with keratin hydrolysates

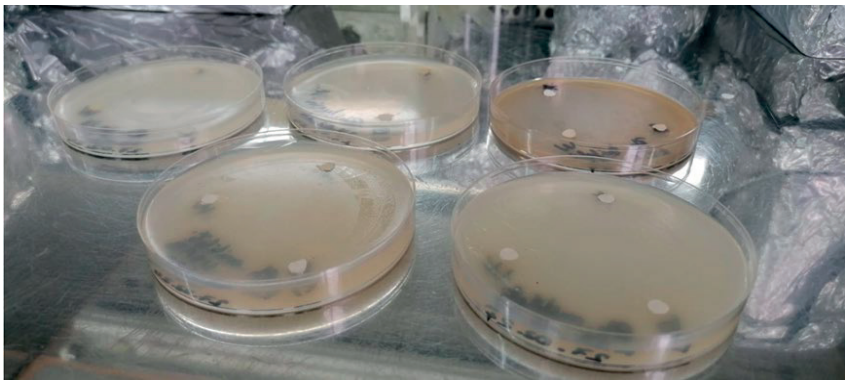

Figure 7. Plates with bacteria of the genus *Rhizobium* grown on YMA medium with discs impregnated with keratin hydrolysates

CONCLUSIONS

The use of wool waste in agriculture is still a relatively new field for Romania, which requires more studies and research.

Knowing the characteristics of wool pellets allows a better management of them, depending on the purpose and the requirements of agricultural crops, and opens new research directions on improving their fertilizer quality.

The results show that:

- the two types of pellets have a similar moisture content, respectively S6 - 9.6% and S9 - 9%;
- S9 pellets, which have a more aerated structure, have a much lower bulk density than S6;
- also, in S9 pellets a higher water absorption rate was observed compared to S6, although the amount of water absorbed had similar values for both samples.

From these results it can be stated that when incorporated into the soil, S9 wool pellets will provide better aeration and lighter soil structure, which is beneficial for plant root growth, but will disintegrate faster which may influence the time of nutrient release into the soil.

In terms of the possibilities of enriching wool pellets with beneficial bacteria it has been observed that the keratin hydrolysates obtained from the wool pellets do not inhibit the growth of the microorganisms tested. The hydrolysates obtained can be used for the development of bacteria of the *Bacillus* and *Rhizobium* genera, which can be applied to agricultural crops to maintain plant health, improve agricultural yields and restore soil biodiversity.

ACKNOWLEDGEMENTS

This work is financed by Ministry of Research, Innovation and Digitalization through Program 1 - Development of the national research-development system, Subprogram 1.2 - Institutional performance - Projects for financing excellence in RDI, Contract no. 1PFE/30.12.2021,, and by NUCLEU programme contr. 9N/01.01.2023, project PN 23 04 02 02

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