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Mats Made from Recycled Tyre Rubber and Polyurethane for Improving Growth Performance in Buffalo Farms

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Abstract: This study focuses on anti-trauma mats designed for buffaloes' comfort, using as raw materials rubber powder from end-of-life tyres (ELTs) and an isocyanate-based polyurethane resin binder. The first part of the study focused on mat formulation. Whilst it was possible to select a unique combination of raw materials and design features, it was necessary to investigate the relationship between three critical parameters affecting mat consistency and therefore buffalo comfort: binder quantity, mat thickness, and desired final mat density (bulk). In order to quantitatively assess the variation in hardness, various combinations were investigated within well-defined ranges based on the relevant literature. The results obtained from nine selected combinations indicate that increases in the three critical parameters do not induce a real phase transition in the final product consistency, although the hardness suggests an increasing trend. The mats consistently exhibited a moderately soft/hard consistency, offering environmental benefits in terms of increased rubber usage and potentially reduced chemical binder, depending on the desired thickness. The selected mixture showed excellent resistance to heavy chemical loads, suggesting reliability for frequent cleaning operations. The second part of the study involved field trials of the mats with calves. This involved monitoring their weight gain and appetite levels over a 90-day period. The results showed excellent growth performance compared to uncoated grids (i.e., weight gain was approximately 20% higher at the end of the observation period); this was similar to that achieved with the use of straw bedding. However, compared to straw bedding, the mats (i) exhibit long-term durability, with no signs of wear from washing or trampling over the months of the trial, (ii) allow for quick and efficient cleaning, and (iii) enable companies to save on labour, material (straw), and waste disposal costs, while maintaining (or even improving) the same welfare levels associated with the use of straw.

Keywords: animal welfare; buffaloes; cattle; chemical stress; end-of-life tyres; hardness; mats; mechanical stress; polyurethane; rubber; Shore A

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1. Introduction

In recent times, the threat of climate change has prompted significant investment by national and international governments in efforts to mitigate the environmental impacts and emissions associated with the consumption of fossil fuels and raw materials [1–3]. In this context, in the integration of state-of-the-art intelligent technologies which allow for real-time monitoring of energy and operational parameters for comprehensive insights into energy-consuming systems [4–11], and the study of innovative processes and technologies for reclaiming organic and inorganic materials from waste streams [12–19], they play a key role. The upgrading of materials derived from different waste sources has been emphasised as a strategic way of achieving sustainability goals and promoting circular economies [10,20,21]. This process involves various routes, including energy production, establishing new markets for by-products, reintroduction into production cycles as raw materials, or transformation and manipulation to create new products for diverse markets [10,21–24]. Within the broader framework of circular economy and waste management policies, the present study focuses on the utilisation of wastes with high environmental impact [25], specifically end-of-life tyres (ELTs), to improve the welfare of animals that are integral to the Mediterranean diet [26,27]—buffaloes.

Buffalo farming is an important industry sector in Italy, with the most significant number of livestock residing in the Campania region [28], where this practice centres around the production of Mozzarella di Bufala Campana DOP. This is often regarded as the "white gold" of the Mediterranean diet [29] and is ranked the fourth most exported Italian DOP product in 2020 [30]. The Campania region houses over 400,000 cattle units, which accounts for more than 75% of the national buffalo stock [31]. Nevertheless, buffalo husbandry is of great socio-economic significance worldwide; indeed, there are over 204 million head all over the world [32]. A reason for the strategic socio-economic role they play can be found in the ecological niche they occupy; in fact, they show high adaptability to hot-wet environments in which other ruminants are not able to thrive, and this is very important because it contributes to the livelihoods of populations living in rural areas [26,32,33]. Therefore, the application of new technologies that can improve the management of animals, their health and welfare conditions, and consequently also the quality of the products that can be obtained from this livestock practice, will play an increasingly crucial role. Since buffaloes are characterised by a peculiar physical structure, which predisposes them to joint overload and limits their adaptability to artificial floors that differ significantly from their natural grazing environments, particular housing configurations may not guarantee optimal comfort for these animals [34–38]. On buffalo farms, there are usually two commonly used types of flooring, namely: metal or concrete grids without coverings, or soft bedding (e.g., straw). The adoption of one type rather than another is related to a farm management choice made by the farmer [39]. In fact, while grids without coverings allow for more practical washing of resting environments, they may be less preferable in terms of animal health, because they are generally associated with higher levels of articular injury and postural changes, and lower levels of cattle cleanliness [40]. On the other hand, straw bedding is often preferred because it is much more comfortable for the cattle [41–43]. However, straw is also more challenging to manage because, in order to maintain specific sanitation standards, it cannot be washed, and instead must be continually replaced with new, clean bedding. This requires more effort to be made by operators (and generates more waste to be disposed of).

Replacing the flooring of resting areas for these ruminants with more comfortable flooring could be an effective first approach to improving their welfare conditions, as has already been demonstrated in other case studies involving cows [44–47]. Indeed, improving the living conditions of buffaloes translates into numerous benefits, including a reduction in the occurrence of joint diseases, an increase in milk yield and milk quality, and an improvement in well-being as a result of providing hygienic resting spaces [36–38,48,49].

When it comes to tyres, their rubber composition consists of high-value polymers with persistent chemical and physical properties that withstand alteration even during recycling phases [50,51]. While this makes tyres highly non-degradable, requiring special disposal methods, it also encourages industry stakeholders to develop strategies that exploit the vast potential of resources that can be recovered from this waste, particularly rubber [52–55]. For the disposal of ELTs, currently two major disposal pathways are adopted [50,56]: (i) recycling, which results in the recovery of specific materials; (ii) energy recovery in specialised facilities, mainly cement plants. During the recycling phase, ELTs are sent to specific plants where, through mechanical shredding at room temperature, they are reduced into increasingly smaller pieces until the three components of the tyre are separated [57–59]: rubber, steel, and textile fibres. For the rubber, fragments of different mesh can be obtained for it, each of these being more or less suitable in relation to potential uses [25,60]. The shredded material has a grain size of between 300 and 50 mm, and is usually used for light filling, drainage, and insulation of civil engineering operations, such as anti-shock paving [54]. Chips have grain sizes of between 50 and 10 mm, and are usually used for common constructions, such as road pavement substrates and sidewalk footings [58]. Granules have a grain size of between 10 and 2 mm, and are usually involved in the manufacturing of various products, such as: floor tiles, roofing materials, floor and wall sound insulation products, road signs, safety surfaces, asphalts, road curbs and barriers, impact dampers, and playground surfaces [61]. Finally, rubber powder has a grain size of less than 2 mm and it is usually reused to produce paints, sprays, electrical cables, automotive parts, sealants, coatings, and flooring [52].

The purpose of this study was to reuse a fraction of ground tyre rubber as raw material to make innovative anti-trauma mats to be employed as alternative flooring in buffalo housing areas. The suggested use of the mats is that they should be permanently installed in resting stalls. They are expected to last a number of years before they need to be replaced, thus bringing many additional opportunities to the companies involved. The presence of rubber mats in cattle husbandry practices has already been demonstrated in terms of its ability to reduce the possibility of hoof damage or of cattle slipping on concrete [62]. This led to a reduction in cases of lameness (usually very common among livestock) and in the costs related to veterinary care [62–65]. In addition to improved health, the presence of rubber mats ensured better hygiene conditions and improved mobility for the livestock; not only that, but the improvement in the overall comfort of the livestock was also positively correlated to the amount and quality of produced milk and to the fertility rate of the livestock [27,44,45]. Of all the ground tyre rubber mesh categories, for the production of the mats we selected the finest one because its grain size offered the possibility of more effective mixing with a proper adhesive and allowed us to produce a more homogeneous product in terms of density and texture.

Regarding the binder to be used, there are several types of industrial resins commercially available that may either be more or less suitable for the purpose. Since in this specific case there was a need to bind rubber powders derived from the dismantling of ELTs, we decided to investigate which type might better suit the context at hand. In the scientific literature there are several examples of rubber powder bound with resins, and each combination is specific with respect to the application of the final product. In many cases, rubber was mixed with epoxy resin for the purpose of enhancing the hardness of the aggregate [66,67]; in other cases, polyester resins were used to supplement rubber from ELTs in the preparation of mortars in order to modify their mechanical properties in construction applications [68]. In the construction industry, the possibility of integrating silica particles into the mixture of rubber and resin was also tested, in order to improve the performance of cement products based on the use of rubber from ELTs [69]; on the other hand, polyurethane resins were frequently used with rubber from ELTs for the production of panels and pads with high sound-absorbing properties [61,70]. Among the various binders applicable with rubber, polyurethane resins were the ones that best testified to the possibility of bringing flexibility and softness characteristics to the end product [71].

The results presented in this paper are part of two research and development projects called MELT and BIOWELT, which have been funded, respectively, by T-Cycle Industries S.r.l. and the Campania Region. Both projects are entirely dedicated to the welfare of buffaloes and result from the desire to find alternative pathways for the re-use of material fractions derived from the dismantling of ELTs, which would lead to the realisation of products with high economic and environmental sustainability value; in this specific case, resting mats. The innovative aspect of this study is related to the desire to selectively focus on the welfare of a species—the buffalo—which, despite being of such socio-economic importance locally and globally, has received less attention from the scientific community than other bovine species. The following sections describe the stages in the realisation of the prototypes and the experimental results obtained from the physico-chemical tests carried out on the manufactured products. Furthermore, based on the results obtained from the mats tests, part of the research involved conducting a first field trial to assess the growth performance of the animals with the new rubber mats (in terms of weight gain and appetite) compared to the use of conventional farm flooring (grid or straw bedding), along with the viability of using the proposed facilities.

2. Materials and Methods

2.1. Defining Benchmarks and Features for Prototype Construction

As anticipated, the main objective of these projects was to reintroduce a fraction of ELTs as raw material in a production cycle aimed at the manufacture of a mat that could be easily used as a replacement flooring in buffalo farms, and that must be able to improve the general comfort conditions of the animal, not only from the point of view of greater comfort of movement, but also for more efficient maintenance of the hygiene and sanitary conditions of the animal. To produce the mats, the basic idea was to recycle selected ELTs rubber waste and fuse it with a suitably selected binder according to a specific design pattern.

In order to define the characteristics of the prototype to be tested, we first analysed which parameters were to be considered most relevant in the construction process. The parameters most represented in the relevant literature [71–79] about this type of products and these specific applications can be summarised and divided into two categories: one dealing with the layout of the mat and the other with its "texture".

2.1.1. Prototypes Layout Features

The first category includes the following:

- The shape of the mat, which can be either square or rectangular;
- The type of surface, which can be flat or raised, providing more or less friction for the hooves, and more or less stability in terms of the risk of slipping;
- The type of base, i.e., the design of the lower surface of the mat, which determines the way in which it is anchored to the underlying surface (smooth, studded, grooved);
- The shape of the edges, which can be smooth and regular, or interlocking (jigsaw);
- The colour of the mat, which can be similar to the natural colour of tyres or coloured with additives to simulate a more natural environment (e.g., dark green).

By varying these characteristics, one could potentially obtain a wide matrix of combinations, not all of which are obviously suitable for the same contexts. As these features relate to the appearance of the mat and depend on the characteristics of the environment in which it is to be used, it was decided to define and use one single combination of these features for all the prototypes tested. Specifically, the mats were intended to be installed in the cattle resting areas, consisting of individual square stalls with a metal grid at the bottom to facilitate washing operations. It was therefore decided to use the following layout: square section (1 m × 1 m), with flat and rough upper surface to ensure proper grip for the buffalo's hooves and reduce the risk of slippering; presence of smooth and regular side edges; grooves on the lower interface to facilitate attachment to the metal grids; as for the colour, it was decided—at this stage—to leave the natural colour of the tyres without adding any other aesthetic additives.

2.1.2. Prototypes Texture Features

The second category includes the following:

- The final polymeric <u>density of the mat</u>, which depends on the properties of the materials used for its preparation and its dimension;
- The <u>thickness of the mat</u>, which together with the density represents another crucial structural parameter that can result in greater or less comfort for the animals;

- Type and quantity of binder, i.e., the resinous adhesive substance added to the rubber powder to make the material compact and compressible.

As for the layout features, there is the possibility of obtaining a multitude of combinations, which could lead to increasing or decreasing the "softness" of the final product. In this study, the purpose of the mat was to be an anti-trauma support that could be a good compromise between softness (when the animals are lying down at rest) and hardness (when they are lowering/raising and walking from one point to another). Narrowing down the field to rubber mats with this specific consistency, we reviewed the most common values and preparation recommendations found in the technical literature for similar applications inter alia [45,46,80,81], which can be briefly summarised as follows:

a. Use of rubber fragments from ELTs with a grain size in the range of 0.8 to 3 mm. In this study, we selected for all of the prototypes the finest rubber fraction, i.e., rubber powder with a particle size $\leq$ 3 mm. The raw material was supplied directly by the company TCycle industries srl, in which, after thorough washing of the ELTs, dedicated industrial equipment performs a dismantling protocol characterised by the following steps: coarse shredding, 2 stages of granulation at increasingly finer cuts, purification of the granules from textile fibres and metal parts, and finally sieving into the various fractions with different grain size. The ELTs granules used in this study for the production of the mats were characterised by a density of 0.487 kg/dm^3, which is in line with similar studies on this category of ELT waste [82]. Rubber granule density is typically determined by the manufacturer using simple gravimetric measurement. The protocol involves measuring the mass of granules filling vessels of known volume and calculating the ratio between the weighed mass and the filled volume. The outcome of this operation is significant because, as explained below, it comes into play when the moulding machinery must be programmed to extract the correct amount of product to produce the final mat, given a defined final bulk density.
b. Before feeding the granules into the mould, they must be thoroughly mixed with the chemical substances required to aggregate/enrich the final product, such as binders, colouring agents, reaction catalysts, etc. For this type of product, the use of an isocyanate-based adhesive was suggested, in varying percentages in the range 5–8% by weight of the total mat mass. Generally, the higher the percentage of binder, the greater the hardness of the mat.
Here, the choice of binder category fell precisely on polyurethane resins with a low isocyanate content. For all of the prototypes we used the chemical binder V-PUR 126 (Vervit® srl, Trento, Italy). This is a one-component polyurethane that reacts with humidity and has very good elasticity. It is normally used to agglomerate SBR (styrene-butadiene rubber) or EPDM (ethylene-propylene-diene monomer) rubber granules in hot presses or at room temperature. This category of adhesives is particularly suitable for this purpose because it also performs better in terms of surface chemical interactions with ELT rubber granules [83], as well as being commercially available and economically competitive. Specific chemical composition properties of V-PUR 126 are given in Table 1.
c. Overall density of the mat (bulk) should stay in the range of 0.8 to 0.9 kg/dm^3. It plays a very important role because it affects the hardness and deformability of the mat.
d. In this study, knowing the specific density of the rubber granules, the density of the binder, and the shape and size each mat should have had (thus the volume occupied), it was sufficient to enter the value of the desired final bulk density for each prototype on the moulding machinery so that it would automatically pick up the amount of raw materials to be combined for the production of the mat. Thickness of the mat in the range 20–30 mm, corresponding to an approximate total volume in the range 0.020–0.030 m^3 (length and width of the mat being fixed at 1 m $\times$ 1 m—see Section 2.1.1).
e. Addition of a small fraction of water (2% by weight) in the rubber/polyurethane mixture to speed up the catalysing reaction.

Table 1. Properties of the product V-PUR 126 (Vervit®).

Feature	Value
Basis	Polyurethane
Appearance	Liquid
Colour	Straw yellow
NCO content	12%
Viscosity, 20 °C (Type RVTD spindle n°2 speed 50)	4000 mPa · s
Density, 20 °C	1.18 g/cm^3
Flammable	No

While only one single combination was chosen for the type of rubber and binder for all the mats, we decided to vary the other three texture parameters (density of the mat, thickness of the mat, and amount of binder) within the suggested ranges, in order to investigate how the different combinations interacted with each other and affected the texture of the carpet (and thus the comfort desired for the buffaloes). In particular, as will be better described in Section 2.3, the experimental setup involved the manufacturing of several prototype mats, each corresponding to a different combination of texture parameters, followed by the execution of physical and chemical tests on each of them.

2.2. Moulding Machinery

As the practical realisation of the mats required the use of moulding presses with industrial performance, it was decided to employ a machine specifically designed for this type of work. In particular, it was used an electric moulding press manufactured by Salvadori s.r.l. (model MOULDING PRESS 60T, Figure 1), which is properly specialised in the production of anti-trauma flooring using rubber granules obtained from the shredding of waste tyres as the raw material. The machine was equipped with two moulds (of different shapes and sizes) and it created the product using compression coupled with mould heating.

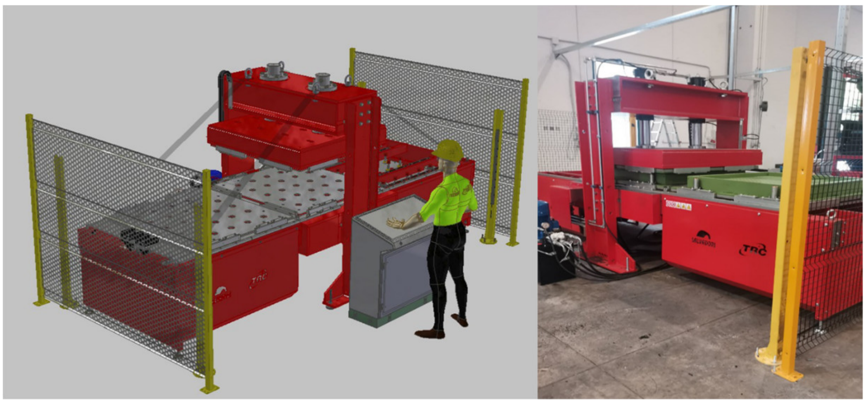

Figure 1. Moulding Press 60T (Salvadori®): rendering provided by the manufacturer (**left**) and the machine installed in the T-Cycle company (**right**).

2.3. Experimental Setup #1: Hardness and Chemical Resistance Tests

As anticipated at the end of Section 2.1.2, according to what was reported in the literature about similar composite materials [71–79], the prototype mats were tested with respect to two physical and chemical parameters considered to be crucial in relation to the intended use of the mat, i.e., hardness (for the consistency) and resistance to chemical agents. The experimental analyses performed aim to answer three questions related to the manufacturing process of the mat. Firstly, whether the process produces a structurally homogeneous product. Secondly, how different combinations of the three texture parameters (final mat density, binder fraction used, and mat thickness) affect the consistency of the mat for use with buffaloes. Thirdly, whether the product is resistant to animal secretions and industrial detergents used to ensure hygiene and animal well-being.

Lastly, considering the results obtained from the physico-chemical tests on the mats, we conducted a preliminary in-farm experiment aimed at assessing the growth performance of the animals (details in Section 2.3).

2.3.1. Hardness Shore A Tests

Shore is one of the static mechanical tests that can be used to measure the hardness of plastic and rubber materials, and it is usually measured using a Shore hardness tester. Tyres, shoe soles, and gaskets are just a few examples, but it is applicable to all polymers, elastomers, rubbers, and gel-like materials. There are different classes of hardness against which Shore can be analysed, depending on the type of object and its specific function [77,84–86]. In our specific case, since the mats are designed to provide a good compromise between structural support for treading and softness (when the buffaloes are standing vs. when they are lying down and at rest), it was decided to test against the Shore A category, which is the one that relates to intermediate structures identified as medium soft/medium hard [71,72,77]. For the measurements, we used a portable Shore A durometer with digital display, with a measuring range of 0° to 100° (against a scale of degrees of hardness) and an accuracy of ±0.1° (Figure 2a).

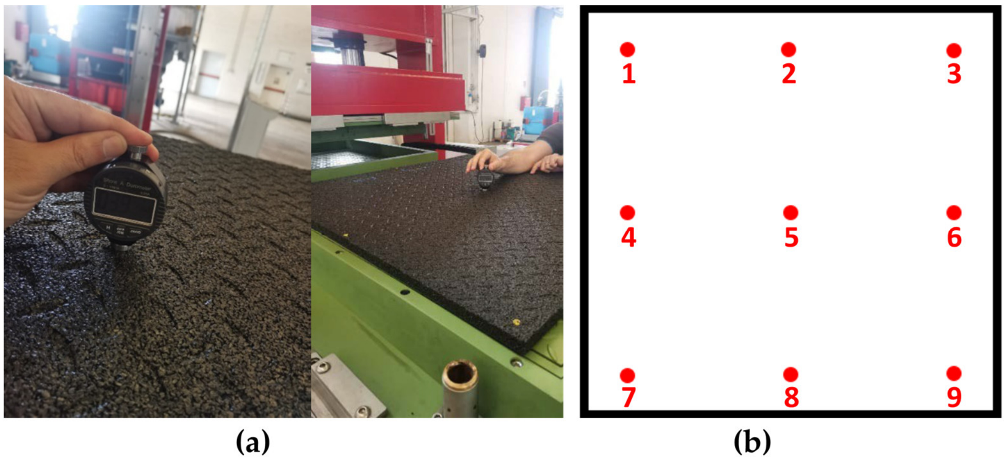

Figure 2. Some photos of the Shore measurement procedure on a sample mat (**a**) and grid pattern for the measurement of Shore A (**b**).

As already discussed in Section 2.1.2, the higher/lower softness of these type of materials (rubber and polyurethane resins) depends very much on the three texture parameters, i.e., density of the mat, thickness, and amount of binder. Thus, we decided to produce samples with different combinations of these three parameters, to assess the hardness achievable for each combination and the relationship between these variables. Overall, the reference values for the combinations are as follows:

Binder percentage equal to 5%–6%–7%–8%;
Final mat density equal to 0.80 kg/dm^3–0.85 kg/dm^3–0.90 kg/dm^3;
Thickness equal to 20 mm–25 mm–30 mm.

The tests were carried out on specific points of the mat, identified through a grid (Figure 2b), with the aim of obtaining an average result as representative as possible of the entire structure of the mat, and identifying any inhomogeneities in the moulding phase. In detail, 4 tests were carried out on a total of 9 mat combinations in order to progressively evaluate the effects that could be obtained by varying only one parameter at a time. Table 2 summarises the mat combinations created for each test with their specific characteristics in terms of density, binder percentage, and thickness.

Table 2. Experimental setup of Shore A hardness tests, with samples ID and features of each sample.

TEST	Sample ID	Final Density (kg/dm^3)	Binder Fraction (%$_w$)	Thickness (mm)
TEST I **Density 0.8 kg/dm^3** **Thickness 30 mm** **Binder fraction variable**	Mat1	0.80	5	30
	Mat2	0.80	6	30
	Mat3	0.80	7	30
	Mat4	0.80	8	30
TEST II **Density 0.8 kg/dm^3** **Thickness variable** **Binder fraction 7%$_w$**	Mat3	0.80	7	30
	Mat5	0.80	7	20
	Mat6	0.80	7	25
TEST III **Density variable** **Thickness 30 mm** **Binder fraction 5%$_w$**	Mat1	0.80	5	30
	Mat7	0.85	5	30
	Mat8	0.90	5	30
TEST IV **Density variable** **Thickness 25 mm** **Binder fraction 7%$_w$**	Mat6	0.80	7	25
	Mat9	0.90	7	25

2.3.2. Statistical Analysis

The durometer results obtained for each mat at each grid point were then analysed using statistical methods to investigate the existence of any significant interactions between the variables. In particular, each mat was evaluated for the distribution of the hardness values over the grid points, then the hardness measurements were analysed in non-aggregated form using descriptive statistics (Pearson and Spearman correlation coefficients), 1-way ANOVA, 2-way ANOVA and Tukey's test, in order to investigate in depth the interactions between the variables included in the tests. Statistical analyses were performed using OriginPro2024 software, while graphical processing was performed using JMP 17 Pro software (SAS Analytics).

2.3.3. Chemical Resistance

In view of the frequent contact between the mats and animal excrement, as well as cleaning operations required to ensure hygiene, the combination of rubber powder and polyurethane used in this study was also tested with respect to contact with highly concentrated acidic and basic reagents. The reagents selected for testing purposes were as follows:

Ammonia NH_3 [32% concentration];
Hydrochloric acid HCl [concentration 3%];
Formic acid HCOOH [concentration 2%];
Hydrogen peroxide H_2O_2 [concentration 30%].

These reagents were selected because they are often present (at low concentrations) in detergent solutions, with special reference to those used in industrial facilities and for sanitation purposes [87,88]. However, the concentration values were raised to levels well above those usually adopted by operators in their everyday practice, in order to create conditions for high chemical stress.

For the chemical tests, the mats were cut with the use of a scalpel to obtain pieces of approximate dimensions 15 × 15 × 10 mm (Figure 3), so that they could easily fit into 50 mL Falcon testing tubes. Three experimental replicates were made for each of the four reagents.

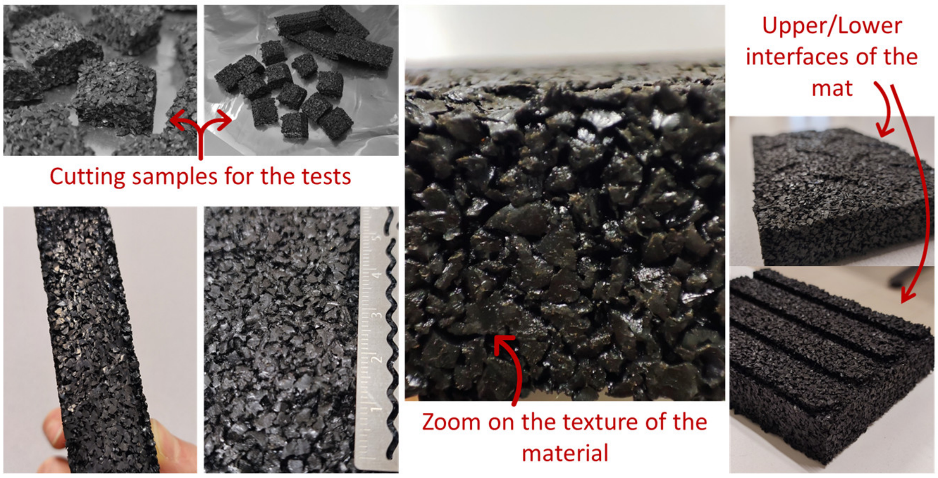

Figure 3. Detail images of the samples used to perform the chemical tests.

These pieces were weighed, in order to gravimetrically assess the potential degradation in terms of mass loss following treatment; an average weight of about 2.7 g was recorded on all pieces. Next, the samples were immerged in 50 mL of each reagent. From this point on, a qualitative assessment of changes in the appearance and/or colour of the samples was made at intervals of 1, 2, and 7 days, by comparing the treated with non-chemically treated samples. At the same time, the pH of the solutions was measured using a pH-meter to verify the potential occurrence of alterations in the chemical solutions.

For the sake of simplicity, with respect to the 9 mat combinations used in the hardness tests, it was decided to perform the chemical tests on only one midrange combination consisting of 7‰$_w$ binder, a thickness of 25 mm, and a final density of 0.8 kg/dm^3. This was possible because the raw materials used in the mat formulations were always the same, while the variations in density and quantity of binder that existed among the 9 combinations did not occur to an extent that would induce noticeable changes in the material's response to a chemical stress.

2.4. Experimental Setup #2: Assessment of the Growth Performance

Following the construction and testing of the mats, a first experiment was conducted in a commercial buffalo farm. In order to prevent any distress to the animals involved, it was decided to simply monitor their weight gain and appetite levels over a period of 90 days in comparison with 5 different floorings. As subjects, it was decided to evaluate the performance of buffalo calves from the 3rd day of life, opting for healthy calves that had no pathologies at birth that could affect growth or nutrient absorption. Three calves were monitored for each flooring to increase the representativeness of the data. The study involved a total of 15 subjects, each housed in a personal stall.

2.4.1. Floorings

Specifically, two conventional floorings (metal grid without coating; straw bedding flooring) were compared with 3 rubber mats having the same values of thickness and binder percentage (25 mm and 7‰$_w$, respectively) and 3 different densities:

- Code Mat_0.8: density 0.80 kg/dm^3;
- Code Mat_0.85: density 0.85 kg/dm^3;
- Code Mat_0.9: density 0.90 kg/dm^3.

The choice was made taking into consideration the results obtained from the hardness tests for the purpose of reducing the number of combinations, which would have required too many animals to be involved in the experimentation.

2.4.2. Body Weight Monitoring

The evaluation parameters for this study were body weight gain and appetite levels. For the former, the animals were weighed at three different times: at the beginning of the experiment (age of the animals: 3 days), after 60 days (age of the animals: 63 days), and at the end of the observation period (age of the animals: 93 days). The apparatus used for the measurements was a cage-equipped floor scale for livestock (PCE Instruments Italia, Lucca, Italy; code: PCE-MS AC1.5T-1-100X200-M-sensitivity: 0.5 kg), which is regularly used in the host farm.

2.4.3. Appetite Level Monitoring

Concerning appetite, knowing the specific feeding plan adopted for the calves by the host farm, it was decided to monitor the amount of food rations taken by each calf, measuring any left-over food in the containers and calculating the difference in relation to the initial value of each ration fed.

During the 90-day observation period, the calves were fed two types of food. Specifically, the farm's feeding programme includes the administration of the following:

- Milk (liquid ration), prepared from high-quality powdered milk specific for the feeding of calves (product name: Zoo Latte Benefit, manufactured by COMAZOO soc. coop. a r.l., Brescia, Italy), fed through graduated buckets (0.1 L sensitivity) equipped with rubber teats and intended specifically for use with calves during the first months of life. For detailed information on the milk and its preparation, please refer to the Supplementary Materials ("Report Comazoo", pages 24–25).
- Fodder (solid ration) for the weaning phases of young calves (product name: Svezza Plus I Periodo-M10, manufactured by Mangimificio Iabichella S.p.A., Ragusa, Italy), delivered by means of non-fixed feeders, in order to be able to easily weigh the incoming ration and any residues at the end of the meal. A professional scale with 10 g sensitivity (Model ICP-WP, ISHIDA Co. Ltd., Kyoto, Japan) was used to measure the feed. For detailed information on the feed and nutritional values, please refer to the Supplementary Materials ("Report Iabichella").

Table 3 summarises the nutritional programme adopted by the company.

Table 3. Nutritional programme adopted for calves on the farm where the tests were conducted.

Phases	Ration Size	Number of Rations Per Day
Phase 1 From day 0 to day 9 since the start of trials	2 L of milk	2 (morning and evening)
Phase 2 From day 10 to day 59 since the start of trials	2 L of milk 1 kg of fodder	2 (morning and evening) 1
Phase 3 From day 60 to day 90 since the start of trials	2.5 L of milk 1.5 kg of fodder	2 (morning and evening) 1

3. Results and Discussion

Analyses started by assessing the hardness of the mat, which is a particularly important characteristic for its intended use, i.e., to serve as a paving layer inside farms dedicated to the growth and care of buffaloes. Taking as a reference the hardness scales conventionally reported for the different Shore categories, the characteristics of an anti-trauma mat produced according to the methods indicated in Section 2.1 lead to the generation of a product of intermediate consistency between soft and hard [72,77,85]. Using the experimental setup described in Section 2.3.1, the Shore A hardness analyses conducted on all samples produced the results shown in Table 4.

On a Shore A hardness scale from 0 to 100, the minimum value corresponds to the transition from soft to moderately soft consistency, while the maximum value corresponds to the transition from moderately hard to hard; the central value of 50 roughly corresponds to the transition from moderately soft to moderately hard consistency [72,74,77,85]. As can easily be seen from the aggregated data presented here, all samples exhibited a quite consistent response to the intent to produce a moderately soft/hard mat.

Table 4. Hardness Shore A levels obtained for each mat combination with respect to the 9 set measuring points, cross-reference to the values of the other parameters (mat density, binder fraction, thickness).

Sample ID	Final Density (kg/dm^3)	Binder Fraction ($\%_w$)	Thickness (mm)	Grid Point	Hardness Shore A (°)	Hardness Shore A Value (°) as: Mean ± SD
Mat1	0.8	5	30	Pt.1	35.84	
Mat1	0.8	5	30	Pt.2	33.74	
Mat1	0.8	5	30	Pt.3	35.85	
Mat1	0.8	5	30	Pt.4	34.57	
Mat1	0.8	5	30	Pt.5	34.49	33.5 ± 2.7
Mat1	0.8	5	30	Pt.6	32.08	
Mat1	0.8	5	30	Pt.7	35.88	
Mat1	0.8	5	30	Pt.8	31.03	
Mat1	0.8	5	30	Pt.9	28.01	
Mat2	0.8	6	30	Pt.1	40.89	
Mat2	0.8	6	30	Pt.2	43.48	
Mat2	0.8	6	30	Pt.3	40.83	
Mat2	0.8	6	30	Pt.4	43.67	
Mat2	0.8	6	30	Pt.5	41.95	42.0 ± 1.3
Mat2	0.8	6	30	Pt.6	42.45	
Mat2	0.8	6	30	Pt.7	43.06	
Mat2	0.8	6	30	Pt.8	42.07	
Mat2	0.8	6	30	Pt.9	39.60	
Mat3	0.8	7	30	Pt.1	47.49	
Mat3	0.8	7	30	Pt.2	47.87	
Mat3	0.8	7	30	Pt.3	47.48	
Mat3	0.8	7	30	Pt.4	45.12	
Mat3	0.8	7	30	Pt.5	44.93	46.3 ± 1.5
Mat3	0.8	7	30	Pt.6	44.05	
Mat3	0.8	7	30	Pt.7	45.72	
Mat3	0.8	7	30	Pt.8	45.61	
Mat3	0.8	7	30	Pt.9	48.33	
Mat4	0.8	8	30	Pt.1	42.57	
Mat4	0.8	8	30	Pt.2	40.33	
Mat4	0.8	8	30	Pt.3	42.28	
Mat4	0.8	8	30	Pt.4	39.17	
Mat4	0.8	8	30	Pt.5	39.43	41.2 ± 1.5
Mat4	0.8	8	30	Pt.6	41.38	
Mat4	0.8	8	30	Pt.7	41.12	
Mat4	0.8	8	30	Pt.8	40.68	
Mat4	0.8	8	30	Pt.9	43.84	
Mat5	0.8	7	20	Pt.1	34.04	
Mat5	0.8	7	20	Pt.2	35.01	
Mat5	0.8	7	20	Pt.3	35.98	
Mat5	0.8	7	20	Pt.4	34.54	
Mat5	0.8	7	20	Pt.5	34.57	36.2 ± 3.6
Mat5	0.8	7	20	Pt.6	34.87	
Mat5	0.8	7	20	Pt.7	35.67	
Mat5	0.8	7	20	Pt.8	34.99	
Mat5	0.8	7	20	Pt.9	45.75	

Table 4. *Cont.*

Sample ID	Final Density (kg/dm^3)	Binder Fraction (%$_w$)	Thickness (mm)	Grid Point	Hardness Shore A (°)	Hardness Shore A Value (°) as: Mean ± SD
Mat6	0.8	7	25	Pt.1	38.31	40.0 ± 1.2
Mat6	0.8	7	25	Pt.2	41.12	
Mat6	0.8	7	25	Pt.3	39.75	
Mat6	0.8	7	25	Pt.4	40.89	
Mat6	0.8	7	25	Pt.5	41.91	
Mat6	0.8	7	25	Pt.6	40.15	
Mat6	0.8	7	25	Pt.7	40.00	
Mat6	0.8	7	25	Pt.8	38.29	
Mat6	0.8	7	25	Pt.9	39.58	
Mat7	0.85	5	30	Pt.1	42.27	40.1 ± 7.3
Mat7	0.85	5	30	Pt.2	45.01	
Mat7	0.85	5	30	Pt.3	30.45	
Mat7	0.85	5	30	Pt.4	33.41	
Mat7	0.85	5	30	Pt.5	37.50	
Mat7	0.85	5	30	Pt.6	39.36	
Mat7	0.85	5	30	Pt.7	32.78	
Mat7	0.85	5	30	Pt.8	48.56	
Mat7	0.85	5	30	Pt.9	51.62	
Mat8	0.9	5	30	Pt.1	42.74	43.3 ± 2.4
Mat8	0.9	5	30	Pt.2	41.10	
Mat8	0.9	5	30	Pt.3	43.20	
Mat8	0.9	5	30	Pt.4	42.74	
Mat8	0.9	5	30	Pt.5	42.68	
Mat8	0.9	5	30	Pt.6	42.72	
Mat8	0.9	5	30	Pt.7	41.82	
Mat8	0.9	5	30	Pt.8	43.48	
Mat8	0.9	5	30	Pt.9	49.44	
Mat9	0.9	7	25	Pt.1	42.20	42.3 ± 1.2
Mat9	0.9	7	25	Pt.2	42.83	
Mat9	0.9	7	25	Pt.3	41.16	
Mat9	0.9	7	25	Pt.4	42.04	
Mat9	0.9	7	25	Pt.5	43.57	
Mat9	0.9	7	25	Pt.6	43.59	
Mat9	0.9	7	25	Pt.7	40.50	
Mat9	0.9	7	25	Pt.8	40.83	
Mat9	0.9	7	25	Pt.9	43.53	

A preliminary observation concerns the homogeneity of the mat structure across all points of the previously established measurement grid. The graph in Figure 4 shows the unaggregated hardness measurements for each grid point of each sample. As can be seen, the consistency of the mat across the nine measurement points is quite homogeneous, with the exception of sample Mat7, which gave the most scattered values. This result is probably not due to the combination set for this mat during preparation (final density 0.85 kg/dm^3, binder content 5%, thickness 30 mm), but rather to the method by which the mat is prepared. While the press mechanically selects and prepares the rubber-binder mixture to be pressed according to the instructions given by the operator on the machine's control panel, the spreading and distribution of the mixture on the mould is carried out by specialised technical personnel who spread the mixture manually using appropriate tools. As can be seen from the graph, this manual process can potentially lead to situations of uneven mat texture and represents an area for improvement, where the introduction of an electronically controlled mechanical device could optimise the uniform distribution of material on the mould. Despite this critical aspect, it should be noted that the hardness differences observed for sample Mat7 are not significant enough to alter its consistency, which remains close to the intermediate range of the transition from soft to hard.

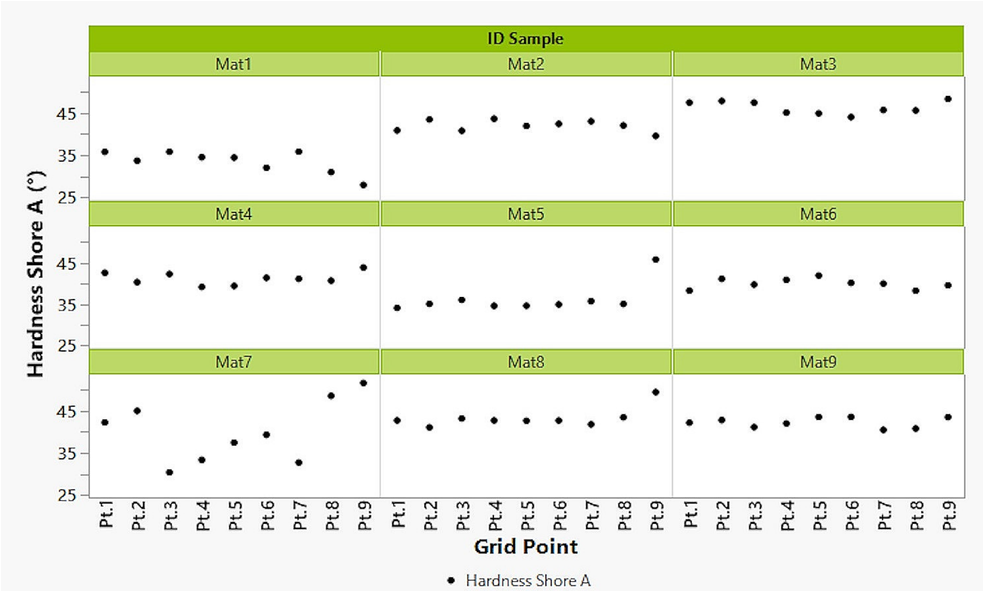

Figure 4. Hardness level distribution for each sample (the grid is reported in Figure 2).

The second question that needs to be answered is how the modification of the three process variables affects product consistency. Figure 5 shows the distribution of the non-aggregated hardness data for each sample with respect to the 4 tests performed.

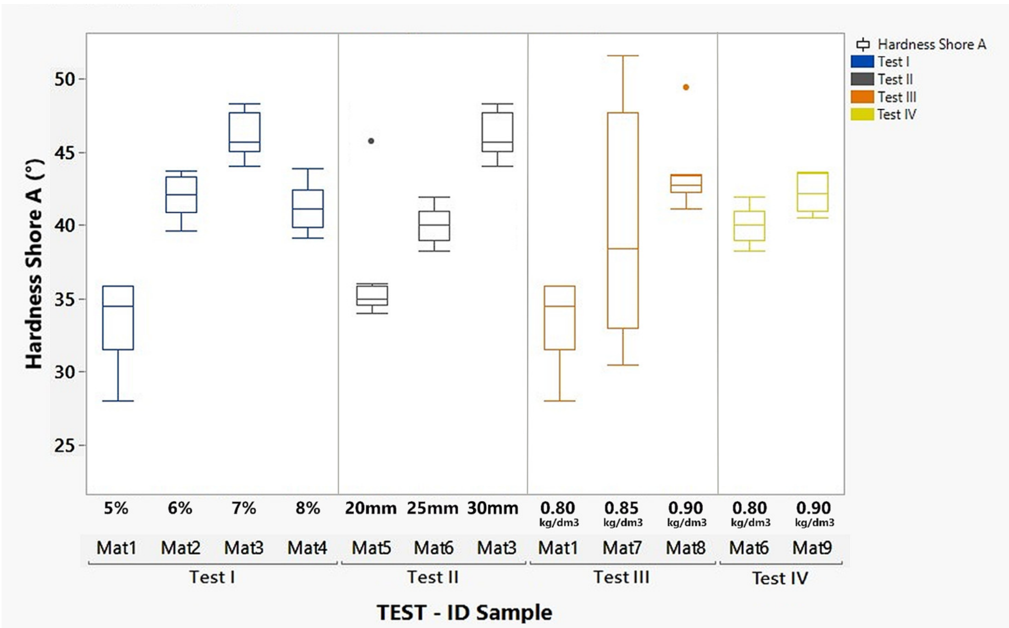

Figure 5. Hardness Shore A levels for each prototype through the four combination tests conducted. The samples are arranged on the graph for each test in ascending order of their respective variables: binder fraction for Test I; thickness for Test II; final mat density for Tests III and IV.

From the graph, it can be seen that the hardness values of the mats appear to be directly proportional to the three parameters, with the exception of Mat4 prototype, which appears to have hardness values that are not in line with the increasing trend of Mat1, Mat2 and Mat3 samples. To accurately determine the statistical meaning of the results obtained for Mat4, it would be necessary to investigate what happens at higher binder concentrations. Sułkowski, Danch, Moczyński, Radoń, Sułkowska, and Borek [72] report results in terms of Shore A obtained on a composite material made with rubber powder and Chemolan binder based on isocyanates of different composition. The experiments showed that a binder percentage of 10% corresponds to an approximate hardness of 60°. They also observed a tendency for hardness to increase as the binder percentage increased, which is in line with the results observed in this study. Revelo, Correa, Aguilar, and Colorado [71] made flexible tiles intended for a different use, but still made with ELTs derived rubber at different grain sizes and a polyurethane adhesive in different percentages; by varying its concentration in the 5–9% range and measuring the hardness of the samples with a scleroscope, they again observed the same result, namely that the hardness of the material increased with increasing resin content, where it appeared to be unaffected by varying rubber grain sizes.

However, since here nine measurements were taken for each mat, it was interesting to investigate whether the average hardness values obtained for each sample or parameter showed statistically significant differences, hence ensuring that the evaluations were most representative. The one-way and two-way ANOVA studies and Tukey's test performed on the data provided the following results (at a significance level $\alpha = 0.05$): the sample with a thickness of 20 mm (Mat5) had a significantly different average hardness compared to the other two thicknesses, which did not show the same difference between them; for the density, the Tukey's test showed that statistically significant differences were only observed between the values 0.80 and 0.90 kg/dm^3, while the intermediate value of 0.85 kg/dm^3 was not sufficiently different from the other two values (see Supplementary Material—Report S1). Regarding the binder content, the average hardness values observed for the four percentages tested were not significantly different from each other (see Supplementary Material—Report S2). These results were also confirmed by the study of correlation coefficients; in particular, the correlation between hardness and binder fraction/final density/thickness was studied using both a parametric index (Pearson) and a non-parametric index (Spearman). The latter was to take into account the presence of possible outliers in the measurements. The results obtained for these indexes (Table 5) essentially confirmed the conclusions drawn from the other statistical indicators, i.e., that hardness showed a positive correlation with the variables, although not truly significant, or at least the statistical significance is not clearly appreciable within the levels considered for the combinations used in this study. More realistically, significant effects might be observed when dealing with much larger variations in each of the three process variables, which would predictably coincide with consistency transitions in terms of Shore.

Table 5. Matrix of Pearson and Spearman correlation coefficients for the Hardness Shore A levels obtained in the four tests vs. the process variables (final mat density, binder fraction, and mat thickness).

	Hardness	
	Pearson	**Spearman**
Final density	0.24605	0.24932
Binder fraction	0.19372	0.13415
Thickness of the mat	0.26405	0.24865

These results are very important for all concerned with the environmental and economic sustainability of the finished product. Having a greater flexibility in the formulation of the mat without significantly altering its consistency enables two significant benefits to be achieved. The first is an environmental advantage, which is linked to the possibility of making mats that have, for the same weight, less binder and more recycled rubber (more recycling and greater ecological sustainability). With less binder and more recycled rubber, the second direct advantage is the possibility of reducing the production costs per mat linked to the purchase of an external reagent (the binder) without, however, compromising the consistency of the mats (economic sustainability). As a result, the well-being of buffaloes, which is the primary goal of producing these anti-trauma mats, should not be compromised. From an economic standpoint, it is worthwhile to consider that T-Cycle Industries company, which is involved in both MELT and BIOWELT projects, usually extracts around 62% rubber powder from 1 tonne of ELTs, which can be resold on the construction market at a cost of around 130€ per powder tonne (data provided by the company). Substituting the conventional polyurethane binder, employed in the following study, with a biological alternative containing, e.g., lignin could provide added ecological advantages for the company and improve the production process behind the mats. Based on recent research, lignin-based polyurethane is a promising alternative which preserves equal chemical and mechanical performance when compared to non-biological adhesives [89,90].

The third and final experimental question that this study sought to answer was whether the mat produced with this composition would be resistant to chemical attack, given that it would be in frequent contact with animal excrements and would be subjected to washing processes aimed at providing satisfactory hygienic conditions. When it came to testing with chemical reagents, here the results were homogeneous regardless of the type of reagent used. After 7 days of being soaked in the solutions (Figure 6), the samples were taken out of the tubes and left to dry under a fume hood for 24 h, due to the presence of chemical vapours arising from the solution residues. Subsequently, they were left in an oven at 35 °C for 3 days for a more thorough drying. Table 6 summarises the results obtained from the evaluations. Figure 6 shows the samples immersed in the reagents during the experiment.

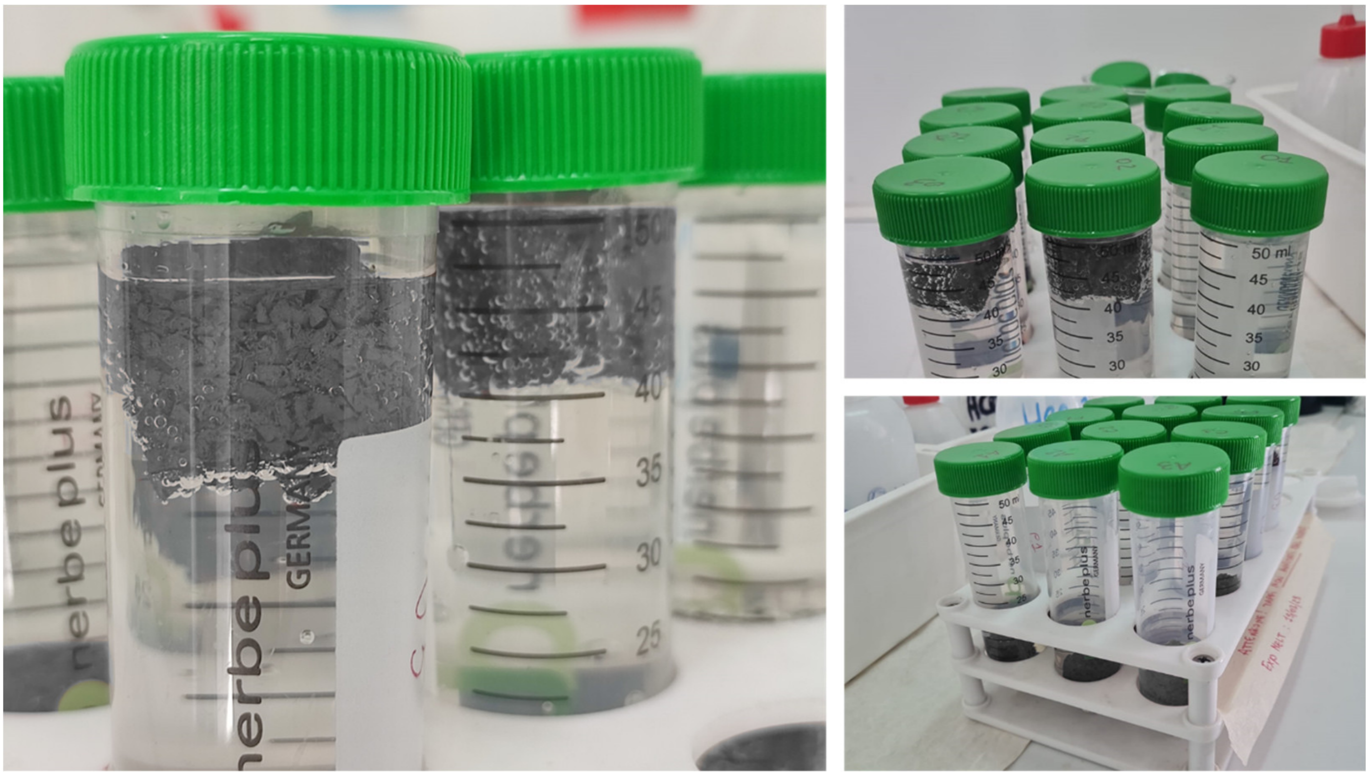

Figure 6. Samples submerged in the different reagents during the chemical stress tests.

Table 6. Summary table of chemical test results. For the colour, "NC" means "no changing".

Reagent	Sample ID	pH of the Reagent				Colour of the Sample		
		Day 0	Day 1	Day 2	Day 7	Day 1	Day 2	Day 7
NH_3	A1	10.0	9.9	9.8	10.1	NC	NC	NC
	A2	10.1	10.0	10.1	9.9	NC	NC	NC
	A3	10.1	10.2	10.0	10.2	NC	NC	NC
HCl	C1	0.1	0.1	0.2	0.0	NC	NC	NC
	C2	0.1	0.0	0.1	0.0	NC	NC	NC
	C3	0.2	0.1	0.0	0.0	NC	NC	NC
HCOOH	F1	2.5	2.5	2.4	2.5	NC	NC	NC
	F2	2.5	2.6	2.5	2.5	NC	NC	NC
	F3	2.5	2.4	2.5	2.6	NC	NC	NC
H_2O_2	O1	4.0	3.9	4.0	4.0	NC	NC	NC
	O2	4.1	4.0	4.1	4.0	NC	NC	NC
	O3	4.1	4.1	4.0	4.0	NC	NC	NC

During the 7 days of tests, there were no qualitative changes in the appearance of the material of which the samples were composed, testifying to high chemical resistance. Confirming the very low reactivity of the samples, it could be noted that the pH also remained unaltered over time. In terms of weight, an average variation of 0.8% was observed over all samples, which is a sufficiently small value to be considered insignificant; such a small variation, in fact, usually falls within the experimental measurement errors. The treated samples are highly porous, and it is safe to assume that—despite being placed to dry for a total time of 4 days—they may still retain within their texture some residue of the chemicals in which they were immersed during the previous 7 days.

Overall, it can be said that the innovative composite material implemented to produce the mats exhibited high and reliable chemical resistance, with its actual composition. The relevant scientific literature confirms the excellent performance of mixtures of rubber powder and polyurethane binders in contact with liquids, indicating that this type of composite material tends to form more interconnected pores, which greatly facilitate the drainage of liquids [78,79]. The same study also evaluated the performance of a mixture of rubber powder and polyurethane from a mechanical point of view (for a poroelastic road surfacing material as an alternative to asphalt), showing that it not only had low slipperiness, but also had excellent resistance to high temperatures and rutting [79]; two very important aspects when considering the thermal comfort that cattle may need in their resting areas on the farm and the obvious trampling to which the mat will be subjected during animal movements.

Following the carpet experiments, we decided to evaluate the growth performance of the buffaloes (mainly the calves) to these different floor coverings in a field trial. To avoid causing any stress to the calves, as mentioned in the Section 2, it was decided to simply monitor their weight gain and appetite levels; the latter by measuring the amount of feed consumed by each subject. Starting from an average weight for all the subjects of 34.2 kg ± 2.9 kg (mean ± standard deviation), Figure 7 shows the average weight gain for each subject with respect to the 5 different floorings adopted. As can be easily noted, the final weights obtained with respect to the 5 floors were: 74.1 kg ± 2.2 kg on the grid, 83.3 kg ± 3.8 kg on Mat_0.8; 88.6 kg ± 2.2 kg on Mat_0.85; 83.1 kg ± 4.7 kg on Mat_0.9; 76.3 kg ± 1.5 kg on straw. On a percentage basis, while the uncovered stalls are those associated with the lowest weight gain over time (+120% in grid stalls compared to day 3), those covered with rubber matting showed rates comparable to those obtained in stalls with straw bedding, i.e.,: +140% in stalls with Mat_0.8, +137% in stalls with Mat_0.85, +146% in stalls with Mat_0.9, and +143% in stalls with straw. All the percentage values were calculated based not on the average weight of all 15 subjects, but on the average weight at day 3 of the calves housed in each of the five flooring types.

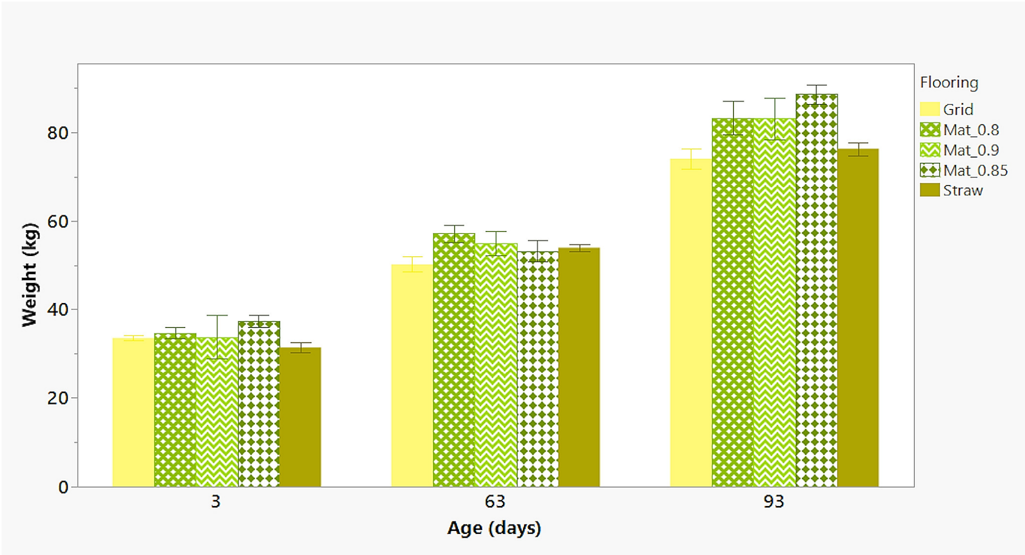

Figure 7. Average weight trend of the calves with the five different floorings.

The results obtained in terms of weight gain are also reflected in the appetite levels (Figure 8), where the good performance of the rubber floorings is witnessed by the consistent results obtained with the three rubber and straw floorings for the intake of liquid (milk) and dry feed (calves fodder). The highly variable pattern observed in Figure 8 for the stalls with grid, on the other hand, are likely to testify to a greater discomfort for the subjects housed in such stalls without floor coverings.

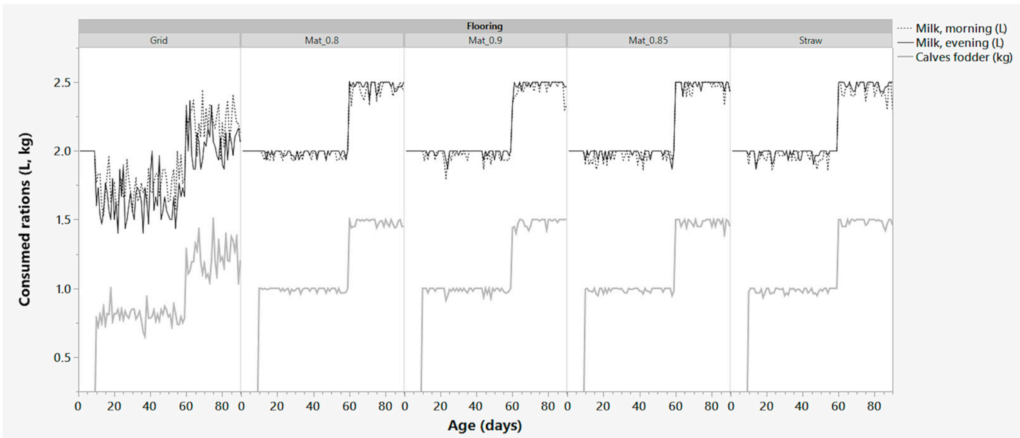

Figure 8. Temporal trend of the liquid and solid rations consumed by the 15 subjects during the day, over an observation period of 90 days from the 3rd day after birth. Data are presented as daily means for each of the five flooring types.

These findings are not entirely unexpected. Indeed, there are already several reports in the scientific literature of suboptimal behavioural and physiological performance associated with solid floorings, such as metal or concrete grids [41,42,80,91,92]. On the other hand, as previously mentioned, it should be noted that the downside of using straw bedding is that it requires a significant commitment in terms of person–hours and material resources to frequently change dirty litters if a certain level of cleanliness and comfort for the animals needs to be maintained. In this context, in order to understand how the rubber mats used here could make a positive contribution to the management of buffalo farms, it is necessary to consider not only the results obtained in terms of growth performance of the calves studied, but also some practical aspects regarding their use. In fact, the mats are installed in the resting pens of the calves, where their intended use is to act as a permanent covering and thus to be trodden on or used whenever the animals need to rest or stay in these pens. The mats designed and tested in this study have been in use at the host farm for several months prior to the writing of this paper and have so far shown no signs of wear from washing or trampling. In addition, their design allows the company's technical staff to rinse the resting areas very quickly and effectively, thus allowing cleaning operations to be scheduled much more frequently than litter changes (which improves hygiene conditions). Finally, the fact that the mats are showing great resistance to wear suggests that they could be used for several years before disposal, justifying the one-off cost of mat installation with the convenience of a short payback time compared to constant straw replacement and disposal (while maintaining the same growth performance and well-being conditions of the reared animals).

In addition, the introduction of rubber matting appears to provide calves with greater thermal comfort compared to conventional flooring, particularly during warmer periods, helping to maintain a cooler temperature. However, this latter aspect requires further investigation (as well as the actual wear resistance over the months), for which future experimental prospects include the installation of intelligent monitoring systems for microclimatic conditions in the presence and absence of mats, and how these conditions affect the physiological responses of the animals.

Finally, a final step will be extending the mat trials to a larger population, this time involving adult buffaloes, for which we will attempt to investigate the relationship between flooring, feeding/behaviour, and the nutritional profile of the milk produced.

4. Conclusions

This study investigates the use of rubber powder from discarded end-of-life tyres (ELTs) to create anti-trauma mats, with the aim of improving buffalo welfare in the context of MELT and BIOWELT projects. Whilst raw material selection and mat design were relatively simple, determining the optimal mat recipe was challenging, particularly in terms of binder content, final density, and thickness. The study investigated the interdependency between these three critical parameters and the impact of their variation on mat consistency by testing various combinations within predefined ranges, based on the scientific literature.

The results showed that changes in binder quantity and mat density did not cause a phase transition in consistency. Instead, the mats consistently exhibited a moderately soft to hard texture across all combinations. The finding presents noteworthy environmental advantages as it has the potential to reduce the use of chemical binders and optimise rubber consumption for mat production, depending on the desired thickness.

Additionally, the rubber powder and V-PUR polyurethane binder mixture exhibited strong resistance to harsh chemical stresses, ensuring reliable and frequent cleaning operations while maintaining high hygiene standards for buffaloes and calves.

Furthermore, taking into account the results of the physico-chemical analyses carried out on the manufactured prototypes, a preliminary test involving calves and three combinations of rubber mats having the same thickness and binder percentage, but different bulk densities, showed that rubber mats, regardless of mat density, can indeed guarantee the same growth performance and feed consumption levels as straw bedding, while allowing more efficient cleaning operations and a promising reduction in operating costs for farmers.

Future research will focus on refining mat recipes and designs, prioritising the introduction of non-invasive smart environmental monitoring systems in resting areas. This initiative aims to explore potential microclimate comfort effects associated with mat implementation. Furthermore, a thorough commercial evaluation will be carried out to assess the advantages of enhancing buffalo living conditions. These efforts are expected to promote innovation in livestock welfare and sustainable agricultural practices, ushering in a new era of environmentally conscious animal husbandry.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/agriengineering6010036/s1, Report S1: Results of ANOVA 2 way performed on Hardness data (here provided in Table 3) versus final density and thickness of the mat; Report S2: Results of ANOVA 1 way performed on Hardness data (here provided in Table 3) versus binder fraction; Report Comazoo; Report Iabichella.

Author Contributions: Conceptualization, A.M. (Antonio Masiello), G.C., A.M. (Antonio Marotta) and C.L.; methodology, A.M. (Antonio Masiello) and M.R.d.C.; validation, A.M. (Antonio Masiello), C.V. and M.R.d.C.; formal analysis, A.M. (Antonio Masiello), A.S., C.V., G.D.S. and M.R.d.C.; investigation, A.M. (Antonio Masiello), A.S., C.V. and C.L.; resources, G.C., A.M. (Antonio Marotta) and C.L.; data curation, A.M. (Antonio Masiello) and M.R.d.C.; writing—original draft preparation, A.M. (Antonio Masiello), M.R.d.C. and C.L.; writing—review and editing, M.R.d.C. and C.L.; visualization, A.M. (Antonio Masiello) and M.R.d.C.; supervision, A.M. (Antonio Masiello), G.C., A.M. (Antonio Marotta), F.D.C. and C.L.; project administration, A.S., C.V., G.C., A.M. (Antonio Marotta), F.D.C. and C.L. All authors have read and agreed to the published version of the manuscript.

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Conflicts of Interest: The authors declare no conflicts of interest.

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