



# MANUAL FOR THE USE OF DEODORISED POULTRY MANURE IN THE BATING PHASE



**LIFE PODEBA PROJECT**

**LIFE10 ENV/IT/000365**

**DELIVERABLE ACTION N° 10**



# MANUAL FOR THE EMPLOY OF DEODORISED POULTRY MANURE IN THE BATING PHASE



LIFE10 ENV/IT/365

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**Tanning** is one of the oldest trades of humanity, with references on the use of leather in cave paintings and archaeological sites worldwide that prove this.

Tanning had an accidental nature in its prehistoric origins, slowly evolving until turning in the Middle Ages into a craft. At the end of the nineteenth century, the first scientific studies were started which, with the industrial revolution and the development of the relevant machinery, derived into the current leather production technology.

### 1. The origins of tanning.

Primitive man hunted wild animals for food; he removed the hides and skins from the dead animal carcass and used them as crude tents, clothing and footwear. The earliest record of the use of leather dates from the Palaeolithic period, cave paintings discovered in caves near Lerida in Spain depict the use of leather clothing. Excavation of Palaeolithic sites has yielded bone tools used for scraping hides and skins to remove hair.

The main problem for the prehistoric man was to avoid the rapid putrefaction of the skins, by first drying and then rubbing them with fat to give them a greater softness. It is assumed that primitive man also discovered accidentally that the smoke from wood fires could preserve the hides and skins, as did softening them with

tannin-containing barks, leaves, twigs and fruits of certain trees and plants.

With the passage of time, they discovered ways to remove the hair using wood ash and burnt limestone and, much later, the capacity of certain minerals (alum) to stabilize the skins obtaining whiter and softer leather thanks to their content in aluminium.

### 2. The industrial processing of the hides and skins.

The tanning process consists in turning a putrescible organic product, fresh animal skin, into a resistant, durable and very nice-looking material that can be used for the manufacture of footwear, leather goods, upholstery, garments, etc. The hides and skins used are mainly those of cattle, sheep, goat, pig and, in a lesser amount, reptiles, birds and fish. Leather processing can start little after slaughtering the animal, but in many cases the hides and skins are stored for a long time, a preservation treatment being necessary to prevent the growing of microorganisms and the associated putrefaction.

The curing process is carried out in collection centres, inside or outside abattoirs, to avoid the putrefaction and loss of quality of the raw material, which requires the time between collection and preparation to be as brief as possible. For this, when the material cannot be processed immediately, it must be cured by one of the available methods: refrigeration(chilling) for short periods, drying (by air or in chamber), salting, dry salting and use of biocides or products to prevent bacterial attack.

Hides and skins are usually cured in abattoirs and sometimes the gatherers/ wholesalers re-preserve them, putting hides and skins in pallets, leaving them in cold areas of the warehouse or refrigerated. The degrees of curing of the skin vary, although currently all the skins are prepared to be able to be transported to any place in the world, which involves undergoing a curing treatment that prevents them from deteriorating over long periods of time and enduring very aggressive transport conditions.

Once in the tannery, if the skins are to be stored for long time, they are kept in refrigerated to avoid deterioration and quality loss.

In the industrial process numerous chemical and mechanical operations are carried out, using different technologies and reactives according to the type of skin that needs to be processed and the

use of the final product, each tannery adapting basic processes to their needs.

The process to obtain finished leather from fresh hides or skins can be divided into multiple steps, which in turn can include four stages:

- Beamhouse operations,
- Tanning,
- Post-tanning
- Finishing.

The Beamhouse operations for a conventional process are the following: soaking, hair removing, liming, fleshing and splitting.

The objective of this stage of the process is to clean the skin, remove the adipose tissue and the hair and adapt the thickness of the skin to the desired value.

The Tanning steps for a conventional process are the following: delimiting, bating, pickling and tanning.

The objective of this stage of the process is to partially degrade the structure of the skin to facilitate the penetration and the subsequent fixing of chemicals, to adjust the pH to the adequate value for tanning, and to stabilize the structure of the collagen by adding tanning agents (the most common ones are chromium salts or vegetable extracts).

Also, for sheepskin there is usually a degreasing step after pickling. After the tanning step the skins are now stable and in this state they are called 'wet-blue', if tanned with chromium, or 'wet-white', if another tanning agent was used.

The Post-tanning steps for a conventional process are the following:

- shaving,
- neutralisation,
- retanning,
- dyeing,
- fatliquoring,
- sammying and drying.

The objective of this stage of the process is to adjust the desired thickness for the skin, achieving the characteristics of fullness and colour, and to bring the skin to a suitable moisture content. In this stage the skin is called 'crust'.

The Finishing stage for a conventional process consists in diverse mechanical operations and/or the application of various products on the surface to give the leather the final texture and appearance desired.

According to the type of starting hide or skin or the final product to be obtained the stages can be carried out in a different way. Consequently, many variations to the conventional process can be found.

The stages of the tanning process are listed in Table 1:

<b>RAW HIDE OR SKINS</b>	
<b>BEAMHOUSE</b>	Sorting-Trimming
	Soaking-Liming
	Fleshing-Pelt splitting
	<b>Bating</b> - Delimiting
<b>TANNING</b>	Degreasing
	Pickling
	Tanning
	Sammying
<b>POST-TANNING</b>	Splitting
	Shaving
	Retanning
	Neutralization
	Dyeing
	Fatliquoring
	Sammying
	Drying
	Staking
<b>FINISHING</b>	Finishing
	Mechanical operations
	Sorting – Packing – Dispatching
<b>FINISHED LEATHER</b>	

Table1. Stages of the tanning process.

### 2.1. Beamhouse operations.

In this stage the initial steps for the processing of skins are carried out from its arrival to the factory until the tanning is carried out. These are chemical processes and mechanical operations to remove all unwanted components from the skin, which are not adequate to obtain the leather, as well as to prepare the structure of collagen for the tanning stage.

Most processes are carried out by immersing the skins in water and adding different chemicals in adequate containers.

The sequence of steps in the beamhouse stage is described below.

### **Soaking.**

The aim is to remove all foreign matter from the skins and to return the skins to the hydration state they were in when fresh. Fresh skins do not require soaking, just a wash to remove blood, dirt and dung.

Cowhides present some problems in soaking, as they are thicker and therefore salt is more adhered to the fibres, which makes it more difficult for water to penetrate. The water that is used for soaking must be free from organic matter, at a temperature of around 25-28 °C and free from ferric salts that can produce stains on the skin.

The products added to the soaking bath are:

- Alkaline products: when the hair is not relevant given that these products break the hydrogen bridges that exist in collagen molecules and subsequently favors dehydration.
- Surfactants: the function of which is to decrease the surface tension of water and thus facilitate water penetration in the skin. They are also able to emulsify the natural fats of the skin.
- Prepared enzyme products: the function of which is to accelerate the soaking of the skin in a controlled way.
- Bactericides: to avoid bacterial growth problems during soaking.

The wastewater of this operation shows high organic matter content and high salinity, which can cause putrefaction problems with bad odors.

### **Liming.**

The hydrated, cleaned skin, some of the proteins of which having been removed in the soaking stage, now passes to the liming stage. This operation aims to remove the epidermis and hair and to loosen the fibrous structure of collagen. To achieve this, it is necessary to use lime. However, in most cases the desired effect is not achieved, so the bath has to be reinforced with sulphides. The skins must not be left for an excessive time in this bath, since otherwise the hair would be attacked by the alkaline bath, which would dissolve hair and would not be able to be separated in the filter.

The pH of the skins after leaving the bath is of about 12.5. After draining the bath the skins are subjected again to some washes.

At present, the hair removed in this process is filtered out from wastewater and the limeliquor is re-circulated. This hair can be used as a fertilizer.

### **Trimming and Fleshing.**

The depilated skins are brought to the fleshing area. The aim of fleshing is to clean the skin by removing the adhering fat and tissues on the underside of the skin to facilitate the penetration of chemicals applied in subsequent stages. This operation is carried out using a fleshing machine. With this process we obtain the skin ready to be split and a by-product called tallow that can be marketed.

The operation ends with a manual retouch to remove what the machine hasn't removed properly.

### **Splitting.**

This operation consists in placing the skin on two cylinders and using a knife, separating horizontally the skin in two layers, one called grain, which will be used to produce leather, and another one called split, which is the flesh part that can be tanned in the same factory, to obtain another external layer or can be applied to other uses.

### **Deliming.**

After the splitting process, the grain layer of the skin and the suitable split undergo the delimiting process.

Delimiting is the process that removes lime and alkaline products from the skin. This way the swelling of the limed skin is achieved.

The lime dissolved in interfibrillary liquids and the one deposited on the fibers (it's only light soluble) can be easily removed using some washes prior to liming. The rest of the lime is removed by adding buffered solutions of ammonium or organic salts, or carbon dioxide. These agents, combined with the alkaline products of the limed skin, provide readily water-soluble products that can be removed by a simple wash. When adding these acids, the pH has to be equal to or greater than 7.5, otherwise the skin would undergo undesired acidic swelling.

### **Bating.**

*This is the process phase object of our project.*

Bating is a peculiar and often unnecessary process in the tanning cycle. Its origin probably goes back a very long time to days when liming was not a

common practice, and when it was found that skins which were badly soiled with dung often gave a softer, stretchier, silkier leather. A century ago the limed and delimed skins were immersed in water, usually in a paddle, at blood heat 37°C, and 3-4 bucketsful of a liquid paste of dog dung and water were added. The skins were run in the liquor for varying lengths of time (1 hour to 24 hours) until they acquired a particularly soft, flaccid and silky handle, as judged by the man in charge. After tannage the leather was found to have a very smooth, flat, flexible grain and to be very soft and stretchy.

Considerable variations in time, temperature and quantities were used for various types of leather. "Puering" was a term specifically used when dog dung was employed, whilst "bating" referred to the use of fowl droppings, which had a somewhat milder action.

Investigations showed that the effect was produced by enzymes (pepsin, trypsin) either from the digestive juices of the animal, which are always present in its dung, or from putrefying bacteria living on the dung. These enzymes, under appropriate conditions of temperature and pH, can digest and dissolve some of the proteins in the skin. In a properly-controlled process they are given only sufficient time for the further removal of unwanted inter-fibrillary proteins, or to modify or weaken those fiber structures which, by binding the collagen fibers tightly together, would cause the grain to be wrinkled and the resultant leather to have no stretch. They also loosen any remaining hair roots, epidermal structure, pigment and fatty cells, so that these can be removed by scudding.

There is the obvious danger of the process getting out of hand and causing overdigestion of the fiber structure, giving thinness, looseness, damaged grain or even holes.

The traditional bating materials used are industrial commercial products based on sterile enzymes (i.e. no live bacteria are present) and are of three main types:

- **PANCREATIC BATES:** These are made by using the digestive enzymes from the pancreatic glands of pigs. These are the glands which secrete enzymes on to the food in the stomach and eventually appear in the dung. These glands can be prepared in a sterile form and are then suitably mixed with fine sawdust (wood flour) and ammonium sulphate or chloride. They appear as a coarse, yellowish, odour-free powder, easy to store and handle.

Optimum pH for activity is usually 8.0-8.5.

- **BACTERIAL BATES:** Appropriate bacteria are encouraged to grow in a suitable solution, which soon becomes full of their digestive enzymes. The living bacteria are then killed by sterilization, and the enzyme solution is prepared as a dry powder in a mixture with wood flour and ammonium salts. Optimum pH for activity is usually 6.0-7.2.
- **MOULD FUNGUS PROTEASES:** These are obtained by growing certain moulds on appropriate media and then separating the mould enzymes. Optimum pH for activity is usually 3.5-5.0. A common additive is a mixture of sodium sulphite and bisulphite.

The relative merits of types of bates are very much a matter of opinion of the individual tanner and depend on the type of leather being made and, in particular, the previous processes carried out.

As commercial products may contain large amounts of inert fillers and buffer salts, it is prudent to check on their enzyme activity. This is judged by the ability to digest alkaline casein (this being a protein available in standard form) and expressed in Lohlein Volhard Units. Weak agents, suitable for hide upholstery leathers, may be rated at 500-1,000 units, shoe upper leathers, 1,000-1,500 units and strong agents, as for glove leathers or glaze kid, 1,500-2,000 units.

In the bating phase method the hides or skins are delimed to a pH of 8.0-8.5 and washed. This is the degree of alkalinity at which most enzymes show greatest digestive power. The goods are then usually paddled or drummed in 300-5000/0 water at 37°C with a 1-2% addition of the powdered enzyme mixture.

When only a slight flattening of the grain or increase in flexibility is required (as in hides for upholstery leather) the time of bating is about one hour, i.e. so short that the enzyme effect can only be judged as a subtle difference in the finished leather. An hour is also common for calf and hides for chrome-tanned shoe upper leathers, but longer times are necessary for sheep and goat, where extreme stretch and suppleness are required, e.g. for gloving.

The digestive action of bating is stopped by producing conditions unsuitable for the enzyme activity, the easiest methods being to cool the skins down to 16°C, to make them more acid, or to commence tannage.

The skins should never be left in the bated condition for more than 1-2 hours, for there is always' the danger of contamination with other putrefying bacteria, which can cause damage even under cool conditions. It should be realised that the enzyme is not "used up" by the process and old bate liquors, if properly used, can be just as strong as fresh ones. They are often more active, due to contamination or infection by other putrefying bacteria.

It is important to realize that to obtain a thoroughly strong bating action it is more important to control (a) the pH of the skin (i.e. the alkalinity, as shown by indicators on a cut cross-section of the skin) (b) the temperature and (c) the time, rather than increase the amount of actual bate given.

The objective of bating is to achieve, using proteolytic enzymes, the de-swelling and relaxation of the skin, while cleaning up any remaining epidermis, globulins, elastin, hair and fat, as a secondary effect, making the grain finer and softer grain.

Traditionally, animal dung, mainly dog and chicken dung would have been used as the source of the enzymes. The digestive tracts of these animals are a rich source of the relevant enzymes. These methods were not favorable because of the unpleasant odor and length of time. For these reasons, in more recent times the enzyme was extracted from pancreas of cattle.

This gland produces a series of active substances able to decompose the proteins and fats.

Bating with such extracts, being unhygienic and hard to control, it has been displaced by artificial bates that simplify the exact dosage and correct control of the operation. **From here the significance of our project. We have solved the problem of odor and hygiene of poultry manure and made an interesting bates agent easy and ecological with no purpose problems.**

By contrast, pancreatic enzymes-based bates are formulated with a large percent of excipients that have a strong ecological impact.

#### Use of the poultry manure in the bating phase

The poultry manure have relaxation properties on the hides and skins in the bating phase because contains the same enzymes present in the conventional products. Passed through a bacteriologic process, the manure is deodorized and his bacterial content downed (deodorized poultry manure, **DPM**).



The skins/hides outgoing of the Deliming phase, at pH 8-8,5, went undergo to the bating.

First time is added clear water at room temperature, 150% on the weight of skins.

Next is added the enzyme and it reacting during 30-40 minutes. The outcome of the relaxation of the skins is hand tested and this time is eventually extended. The use of an imbibent, like a etoxylate fatty-alcohol, is necessary for the distribution of the products in the deep of the hide, and to improve the mechanical cleaning of the impurities.

We have made the same with the DPM.

**Deodorised poultry manure resulted to have an enzyme activity of 1,364 LVU/g over dry weight, a little lower than commercial bating products (1,000-2,000 LVU/g), and it is used in the same way as conventional formulates.**

The difference spotted in the process are due to physical condition of the DPM: it need more time to imbibe with water and set the enzyme free. In the next table a typical recipe of bating with DPM.

150%	WATER 25°C		
<b>0,5-0,7%</b>	DPM (Deodorized Poultry Manure)	<b>30-60'</b>	Relaxed structure
0,2	Ethoxylated fatty alcohol		
	DRAIN		
200	WATER 25°C	5'	
	DRAIN		

Table 2. Standard recipe for bating with DPM

## 2.2. Tanning operations

### Degreasing.

At this stage the natural fat of the skin is removed to facilitate the penetration of reagents and to avoid undesired reactions and stains on the skin. This operation is carried out by direct emulsion of fat in an aqueous medium using surfactants or solvents. When degreasing using surfactants, the process can be improved using lipolytic enzymes that degrade the skin's fat, which facilitates fat emulsion and solubility to reduce its molecule size.

### **Pickling.**

This stage can be considered as a complement of deliming and definitive interruption of the enzymatic effect of the bating process. Also, after pickling the skin is prepared for the tanning operation.

The pickling operation is more important with respect to the subsequent tanning operation, since, if unpickled, the pH of the skin would be high and the salts of the mineral tanning agent would gain higher basicity. In these pH conditions, over-tanning would affect the outer layers, which would make it difficult for the tanning agent to diffuse into the internal layers, thus shrinking the grain layer and precipitating the hydrolyzed mineral agent on it.

In the pickling operation, the skin is treated with acid products that add an important quantity of acids to the skin and at the same time manage to lower its pH to 3-3.5, removing totally the skin's alkali. The acids that are used are: sulphuric acid, formic acid and acetic acid.

Neutral salts are also added to the pickling bath before adding acid so as to be able to prevent the acidic swelling of the collagen. The salt that is normally used is sodium chloride, which is practically not combined with the skin, so its concentration remains almost unchanged in the waste bath. In addition to sodium chloride, it is also possible to use sodium sulphate, sodium formiate and polyphosphates.

### **Tanning.**

The skin, once adequately prepared in previous processes, is subjected to the tanning process in which it turns into leather, namely, the skin becomes rot-proof, stabilizing its protein structure by cross linking collagen chains with the tanning agent by means of chemical bonds.

Due to the wide variety of hides and skins, it is easy to assume that there are many different types of tannages.

All these tannage types can be grouped into:

- Tanning with inorganic products or mineral tanning, using chromium salts, aluminium, iron, titanium, etc.
- Vegetable tanning, using natural vegetable extracts.
- Tanning with organic products like syntans, aldehydes and quinones, sulphochlorinated paraffins and multiple resins.

## **2.3. Post-tanning operations**

### **Sammying.**

Chromium-tanned leather contains between 90-100% of water, but in order for the leather to be adequately split and shaved, it must not contain more than 50-55%.

Leather sammying is easier if the skin has been previously split. This operation is carried out making the leather go through two rollers covered with filter nets. The pressure of the cylinders is transmitted to the leather's fibres and forces them to squeeze the water out, while the filters absorb the water and drain it to the outside.

### **Shaving.**

The leather is passed through a cutting machine subjecting it to a knife that adjusts its final thickness, generating waste called shavings.

### **Re-tanning.**

In the re-tanning stage, one or various tanning products are added to provide the leather with certain qualities that are not easily obtained using only one tanning agent.

There are many types of re-tanning agents that can be grouped as follows:

- Cationic products such as metallic salts like chromium, aluminium, zirconium, organo-chromium and organo-aluminium salts.
- Anionic products such as vegetable extracts like mimosa, quebracho, chestnut, tara, sumac, synthetic substitution products, neutral or acidic auxiliary synthetic products, auxiliary-substitution and vegetable extract-substitution products or mixed blends.
- Resins: anionic, cationic, amphoteric, pre-polymerized, polymerized, monomeric,

urea-based, melamine, dicyandiamide and acrylic resins.

- Various re-tanning agents, like silicate, aldehydes, polyphosphates, tanning oils and fillers of different types.

#### Neutralisation.

The main purposes of neutralisation are to remove the remains of shavings adhered to the skin, to remove unfixed tanning agents and to remove part of the initial acidity of the skin by adding alkaline salts to facilitate the penetration of the dyestuff and fatliquoring products.

#### Dyeing.

Leather dyeing includes a group of operations aiming to confer a certain coloration to the tanned skin, be it superficial, partial or total.

From a chemical point of view, dyes are classified as natural and synthetic; the same way there are vegetable and synthetic tanning agents. The commercial series of dyes gather dyes of very different chemical composition, but of similar dyeing behavior with regard to fastness, penetration power, matching capability, degree of opacity, method of use, etc.

According to these characteristics dyes are classified as: acid dyes, direct dyes, basic dyes, metal-complex dyes and reactive dyes.

#### Fatliquoring.

The fibres of the wet leather move easily since it is a very flexible material; but when the leather dries it can become hard due to the fact that the fibres have dehydrated and have grouped forming a compact substance.

The fatliquoring operation is carried out with the aim to obtain flexible and soft-feeling leather, which is achieved by the incorporation of water soluble or insoluble fats. These maintain the fibres separated and lubricate them so that they can glide off each other.

The greater or lesser degree of flexibility of leather depends on the quantity and type of fat used, which conditions the product that needs to be obtained so that by varying the fat percentages and the combinations of fatliquoring agents different products are obtained.

#### Drying.

Through this operation, the moisture content of the skin is reduced to be able to carry out the finishing operations. Drying can be achieved through different systems.

## 2.4. Finishing operations

The finishing operations include a series of processes aimed to improve the leather's surface appearance, protect it against chemical and mechanical effects, even out color and shine and improve the feel of the leather.

The finish provides resistance to rain, blows, rubbing and any type of external mechanical stress, and at the same time gives the desired appearance to the leather.

Depending on the appearance of the leather's surface and the desired result, the finish application will be different, so if the leather's characteristics are to be highlighted, finer coats will be applied that give it shine and texture but if the leather's imperfections need to be corrected it is necessary to apply thicker coats. Also, pigments are generally added to the finish to even out and adjust the color achieved in the previous stages.

## 3. Product characteristics of the leather bated with new process

The products obtained from DPM bating are tested according to the standard test method:

	DPM (average)	Garment
<b>Tensile strength (N/mm<sup>2</sup>)</b>	22,3	> 15
<b>Elongation percentage</b>	45,9	40-70%
<b>Contraction temperature</b>	>100°C	>100°C
<b>Creases *</b>	No damage	No damage
<b>Strength of grain (elongation at crack mm)</b>	8,2	> 7mm

\* after 100,000 cycles

This leather has the around the same properties of a standard processed product. Furthermore the aspect of the grain, the texture and the visual aspect are very good.

## 4. Environmental consideration

During all the project phases, chemical validation, LCA study and comparisons between garment and footwear products manufactured from tanned hides using deodorised poultry manure in the

bating phase and standard tanned hides were carried out, in order to prove the environmental benefits linked to the use of a by-product (deodorised poultry manure).

The PODEBA environmental benefits can be summarised in the following points:

1. PODEBA demonstrates **a new way to use poultry farm by-product**, solving in part the manure disposal and environmental problems.
2. The odour analysis show a significant reduction in all bad smell indicators' concentrations, demonstrating the **efficacy of the treatment in deodorising poultry manure** (odour was the main limitation in using poultry manure as bating agent).
3. Treated manure **ammonia emission reduction by 80%** in comparison with initial wet laying hen dejection.
4. In agreement with subjective assessment by the tanning experts, the analysis shows a **reduction of olfactive impact in tannery**, in the use of DPM in comparison with first generation DPM.
5. The values obtained in the characterisation of the **deliming/bating baths** in the different tests conducted show that in all of them, there is a **significant reduction in the impact on the environment**, mainly regarding the nitrogen content of the wastewater, measured as TKN/NH<sub>4</sub><sup>+</sup>, and also in the COD, observing an increase in the quantity of the suspended solids, owing to the granular nature of the poultry manure samples.
6. The **LCA study** confirmed the PODEBA environmental benefits, showing that the choice of using DPM instead of standard formulates results in **an overall improvements of the potential environmental impact of the bating process**.
7. **DPM use does not cause trace of oxidation or darkening on metallic parts of our articles.**

8. The **satisfaction of the footwear company** in producing articles using hides bating with deodorised poultry manure is proven.

Aside from the modification of the chemical effluent in the DPM process of bating, need care in regard that the normal chemical products employed in bating phase, are enzymes blended with in organics salts for lower their power. Most of them have a **content of ammonium sulphate between 30-40%. This is not needed to DPM that have an enzymatic power right for employ it. So the environmental impact is lower just before its use.**

All these results, together with the positive effects on leather structure and cleaning surface, indicate that PODEBA bating agent is suitable for tannery applications in all its aspects, especially from the environmental point of view.

## 5. PODEBA leather products

Finally, we report the results obtained when we use the PODEBA bated leather to make upholstery and garment.

We tested many samples of the leather bated with DPM and found the same physical properties of the standard leathers; therefore we expect a similar behaviour in handmade garments.

The commissioned artisan that make footwear, garments and furnitures haven't found significant difference between regular or PODEBA bated leather.

The operations in process of DPM-bating are very similar to the use of the standard product. In any case the final decision on the effective validity of the bating is the hand test of "softness". The smell during the process isn't more annoying. The only relevant difference is the contact time due to swelling problem of the dry DPM.

Finally, the DMP bating agent have a advantageous price. For this reasons we think that the product will have a sure employ in the tanning industries.

