

# Animal fats for non-food uses. A review of technology and critical points

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In this paper some aspects related to the production and the use of animal fats for non-food uses are discussed. The discussion starts with the classification of non-edible animal fats as a consequence of the feedstock quality and of the related potential health risk. The available technologies for fat production and sterilisation are discussed as well as the new problems related to the use in energy production, in oleochemistry and in biodiesel preparation, with a special focus on the reduction/elimination of unwanted contaminants such as metals, phosphorous, sulphur, plastic materials, etc. Some data related changes in concentration during processing of listed contaminants are shown and discussed.

## INTRODUCTION

Non-edible animal fats are produced as unintentional by-product of the food chain. Animal fats are prepared from beef (tallow), pork (lard), chicken and other animals at the end of slaughtering chain for the preparation of food products. Lower grade products are obtained from expired foods or from collective catering waste as well as from the processing of whole dead animals due to illness or accidents.

Until the appearance of TSE (Transmissible Spongiform Encephalopathy) disease animal fats were classified according to their intrinsic quality such as MIU (Moisture, Impurities and Unsaponifiable) criteria, that influenced quality and yield of finished products.

After the publication of Regulation (EC) no. 1774/2002 [1] these non-edible fat products were classified according to the risk for BSE strongly correlated with the anatomic source and with the health state of the animal.

According to the above-mentioned Regulation, non-edible fats were classified in 3 different risk categories, as reported in Table I.

At the time of the publication of this Regulation, Category 1 materials had to be prepared and destroyed in the same authorised processing unit and no possibility for other uses in other places did exist.

Seven years later, when the TSE problem was nearly under control a new Regulation was published (Regulation (CE) 1069/2009) [2]. This new document maintained the fat classification in three categories and allowed the transfer outside the rendering authorized unit and the use of Category 1 and 2 fats after a documented and approved process of heat treatment.

According to the data reported by the European Fat Processors and Renderers Association (EFPPRA) the total annual production of non-edible animal fats in Europe ranged in the period 2009 - 2014 between 2.0 and 2.5 MT/year. Within this total amount 0.7-0.8 MT/year are represented by Cat 1 and Cat 2 animal fats [3].

The main uses of obtained fats are:

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**Table I** - EU classification of non-edible animal fats (from Regulation (EC) no. 1774/2002)

**Category 1** (art. 4): material shall comprise animal by-products of the following description, or any material containing such by-products: (a) all body parts, including hides and skins, of the following animals: (i) animals suspected of being infected by a TSE in accordance with Regulation (EC) No 999/2001 or in which the presence of a TSE has been officially confirmed, (ii) animals killed in the context of TSE eradication measures, (iii) animals other than farmed animals and wild animals, including in particular pet animals, zoo animals and circus animals, (iv) experimental animals as defined by Article 2 of Council Directive 86/609/EEC of 24 November 1986 on the approximation of laws, regulations and administrative provisions of the Member States regarding the protection of animals used for experimental and other scientific purposes (2), and (v) wild animals, when suspected of being infected with diseases communicable to humans or animals; (b) (i) specified risk material, and (ii) where, at the time of disposal, specified risk material has not been removed, entire bodies of dead animals containing specified risk material; (c) products derived from animals to which substances prohibited under Directive 96/22/EC have been administered and products of animal origin containing residues of environmental contaminants and other substances listed in Group B(3) of Annex I to Council Directive 96/23/EC of 29 April 1996 on measures to monitor certain substances and residues thereof in live animals and animal products and repealing Directives 85/358/EEC and 86/469/EEC and Decisions 89/187/EEC and 91/664/EEC (3), if such residues exceed the permitted level laid down by Community legislation or, in the absence thereof, by national legislation; (d) all animal material collected when treating waste water from Category 1 processing plants and other premises in which specified risk material is removed, including screenings, materials from desanding, grease and oil mixtures, sludge and materials removed from drains from those premises, unless such material contains no specified risk material or parts of such material; (e) catering waste from means of transport operating internationally.

**Category 2** (art. 5) material shall comprise animal by-products of the following description, or any material containing such by-products: (a) manure and digestive tract content; (b) all animal materials collected when treating waste water from slaughterhouses other than slaughterhouses covered by Article 4(1)(d) or from Category 2 processing plants, including screenings, materials from desanding, grease and oil mixtures, sludge and materials removed from drains from those premises; (c) products of animal origin containing residues of veterinary drugs and contaminants listed in Group B(1) and (2) of Annex I to Directive 96/23/EC, if such residues exceed the permitted level laid down by Community legislation; (d) products of animal origin, other than Category 1 material, that are imported from non-member countries and, in the course of the inspections provided for in Community legislation, fail to comply with the veterinary requirements for their importation into the Community, unless they are returned or their importation is accepted under restrictions laid down under Community legislation; (e) animals and parts of animals, other than those referred to in Article 4, that die other than by being slaughtered for human consumption, including animals killed to eradicate an epizootic disease.

**Category 3** (art. 6) material shall comprise animal by-products of the following description, or any material containing such by-products: (a) parts of slaughtered animals, which are fit for human consumption in accordance with Community legislation, but are not intended for human consumption for commercial reasons; (b) parts of slaughtered animals, which are rejected as unfit for human consumption but are not affected by any signs of diseases communicable to humans or animals and derive from carcasses that are fit for human consumption in accordance with Community legislation; (c) hides and skins, hooves and horns, pig bristles and feathers originating from animals that are slaughtered in a slaughterhouse, after undergoing ante-mortem inspection, and were fit, as a result of such inspection, for slaughter for human consumption in accordance with Community legislation; (d) blood obtained from animals other than ruminants that are slaughtered in a slaughterhouse, after undergoing antemortem inspection, and were fit, as a result of such inspection, for slaughter for human consumption in accordance with Community legislation; (e) animal by-products derived from the production of products intended for human consumption, including degreased bones and greaves; (f) former foodstuffs of animal origin, or former foodstuffs containing products of animal origin, other than catering waste, which are no longer intended for human consumption for commercial reasons or due to problems of manufacturing or packaging defects or other defects which do not present any risk to humans or animals; (g) raw milk originating from animals that do not show clinical signs of any disease communicable through that product to humans or animals; (h) fish or other sea animals, except sea mammals, caught in the open sea for the purposes of fishmeal production; (i) fresh by-products from fish from plants manufacturing fish products for human consumption; (j) shells, hatchery by-products and cracked egg by-products originating from animals which did not show clinical signs of any disease communicable through that product to humans or animals; (k) blood, hides and skins, hooves, feathers, wool, horns, hair and fur originating from animals that did not show clinical signs of any disease communicable through that product to humans or animals; and (l) catering waste other than as referred to in Article 4(1).

- i)* self-consumption in rendering units to produce electric power, heat and steam [4];
- ii)* as a feedstock for biodiesel production [5];
- iii)* as a chemical intermediate for oleochemistry;
- iv)* as a feed ingredient for cattle and for pets (category 3 only) [6].

About biodiesel production the ratio between Cat. 1 + Cat. 2 vs Cat. 3 as a feedstock changed during the years, following the different possibilities offered by the regulation changes. In 2010 only 40% of cat. 1 + 2 were in use for biodiesel production that became 67% in 2012 and 57% in 2014 [3]. Shifting the use of starting material for biodiesel production has a strong

impact on the preparation of fat before biodiesel production as well as in biodiesel refining before the final use.

The market prices and the available amounts were, more or less, stable during the time, until the appearance of energy uses for natural oils and fats. As one can imagine, the request and consumption for energy purposes is far higher than the one for oleochemical uses. The market price and availability were greatly disturbed by this new factor and prices of good and poor-quality feedstocks raised immediately [7].

Actually all categories of non-edible animal fats are used for energy purposes, both in the neat or in trans-

formed (biodiesel, fatty acids methyl esters) form.

These new (for the energy market) materials posed several different problems for the use in the biodiesel plants and in different engines that have been faced and partly solved during these last years.

Scope of this paper is to provide a complete picture of the production technology, of the composition and of the existing problems related to the use of animal fats for non-classic oleochemical applications. The basis for this discussion comes from a wide monitoring of the international literature and from the direct professional experience of Authors.

## THE PRODUCTION TECHNOLOGY

Animal fats are commonly prepared from animal feedstocks by means of the so called "rendering" process. In few words rendering consists in cooking starting material as it is (wet processing) or after the evaporation of existing water (dry processing), after the size reduction of different anatomic parts and bones.

The separation of the three rough constituents of the feedstock (fat, solid fraction and water) is carried out in the wet rendering by means of decanters and centrifuges, while in the dry process the fat is obtained through water evaporation and pressing of dried feedstock, allowing the separation of fat and solid material.

Temperatures in the wet process are relatively low and, in every case, lower than 100°C. This technology allows obtaining a better quality for both fat and solid material. After cooking (100°C × 1 hour as an indication) the obtained sludge material is sent to a triphasic decanter, where fat, water and solid material are separated. The crude fat is delivered to filtration and drying units, water is sent to the waste water treatment and the solid material is processed with a screw press, in order to recover the remaining fatty material and part of water. Part of unwanted contaminants are removed by means of the removal of the aqueous phase.

On the contrary, the dry process is carried out by means of evaporation and temperatures can reach 130°C. The cooked and dried feedstock is finally processed in a screw press for the separation fat/solids. In this case the fraction of contaminants removed by centrifugation in the wet process remains in the two main fractions.

The recent changes in the fat uses for energy purposes caused a return to the wet processing because of the better quality of the final product.

In Figure 1 and 2 the flow sheet of the two discussed processes are reported.

The rendering activity can be regarded not only as an industrial one, but also as an activity having a strong social impact. In fact, the existence of animal fats rendering units is necessary to make the meat

production chain complete and it has been always used for the elimination of residues generated by the preparation of primary products. Just to provide an overlook of the impact of rendering in the complete meat production chain, we must consider that the proportion of the weight of slaughtered animal used for food purposes and sent to rendering represents 34% in case of swine, 42% in case of beef and 25% in case of poultry, for a total annual amount in Europe of 12 MT delivered to the rendering units [8].

Many years ago, all rendering units were operating far away from towns and villages, because of the strong smell emissions related to the storage and processing of meat by-product. The recent social changes and the expansion of civil installation made houses very close to the rendering units, with big problems related to high duty traffic, trouble and smell.

To solve this big problem, the most recent installations have been designed in order to keep the unit under a light vacuum in tightly closed building. The waste air is extracted by means of high flow vacuum pumps and the waste air is treated in a combustion system or in a biological filter.

These new plants demonstrated to have a negligible impact on the surrounding areas.

The final products of the rendering process are meat-and-bone and animal fat derived from Category 1 and 2 materials and processed animal protein and animal fat from Category 3.

By a combined reading of Regulation (UE) 142/2011 [9] Annex III, Annex IV, the fat can be used as fuel if it meets the following prescription:

- Fat from Cat 1 or Cat 2 must be processed using method 1 to 5 (Annex IV, Chapter II, Section 3 of Regulation (UE) 142/2011 [9])
- Fat from Cat 3 must be processed using method 1 to 5 or method 7 (Annex IV, Chapter II, Section 4 of Regulation (UE) 142/2011 [9])

Cat. 3 fats can be used as a feed for ruminants, with the only condition that total impurities must be lower than 0.15 % (m/m).

- Method 1 to 5 must be used for Cat 1 and 2 if the obtained fat is used for oleochemical or energetic purposes (Annex XIII, Chapter X 3 of Regulation (UE) 142/2011 [9])

Method 1 is mandatory for fertilisers from Cat. 2 meat and bone meal. As fats are not in use as fertilizers this method is not applicable for fats.

The quality of obtained material can vary dramatically, according to the nature of starting material, its storage time and conditions, the presence of microorganisms, insects, etc. For many years the finishing and the storage of bad quality animal fats was a careless operation, but since the use of these products in the energy sector became more and more important procedures necessary to have a stable product have

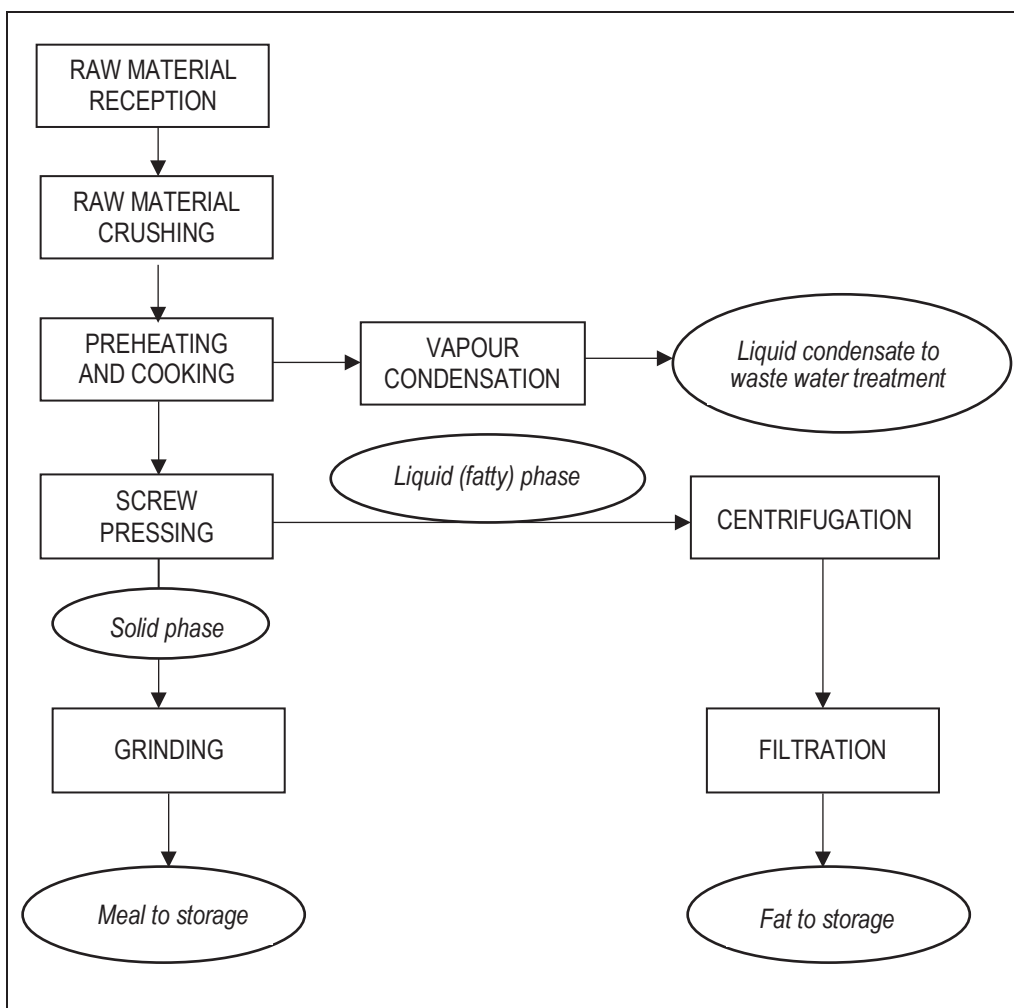


Figure 1 - Flow sheet of dry rendering process (original drawings from Lipitalia 2000 SpA)

been put in place. The minimum required treatment is the removal of all suspended solids and particles as well as the removal of water. If stored without these precautions, animal fats can quickly increase in acidity and in rancidity [10].

### CHEMICAL COMPOSITION

Also, chemical composition is extremely variable, according to the nature of the feedstock. Fat is recovered by rendering from all animal sources (bovine, swine, poultry, fish, etc.) as well as from catering wastes and expired or badly preserved foods. For these reasons a robust knowledge of starting material is necessary to select and separate different starting materials and finished products. A recent fact happened with a sample of animal fat having a Calcium content over each previous experience. At the end of the tracing activity we discovered that this fact was obtained from a batch of battered cuttlefish, still containing the bone. During the early 2000 we participated at an important

European Project, whose name was "Feeding Fats Safety" and we had the chance to collect and to analyse several samples from rendering activity throughout Europe.

These data were published at that time and in Table III a summary of these compositions, is reported. Fish oils are not included [11].

From the reported data the huge distribution of chemical composition can be confirmed, notwithstanding the median and average values are always located far from maximum detected values. Acid value is in average not so high, even in consideration that values are reported as mg KOH/g, representing roughly the double of acidity as % in oleic acid. The concentration in mono and diglycerides reflects this situation and is very important to note that monoglyceride concentration is negligible. This is an important fact in case of physical refining where higher monoglyceride amounts may create problem inside the distillation column. Also, oxidative degradation does not seem to be a problem for industrial applications as well as polymer triglyceride concentration. About the presence of vitamin E and derivatives it is well known that

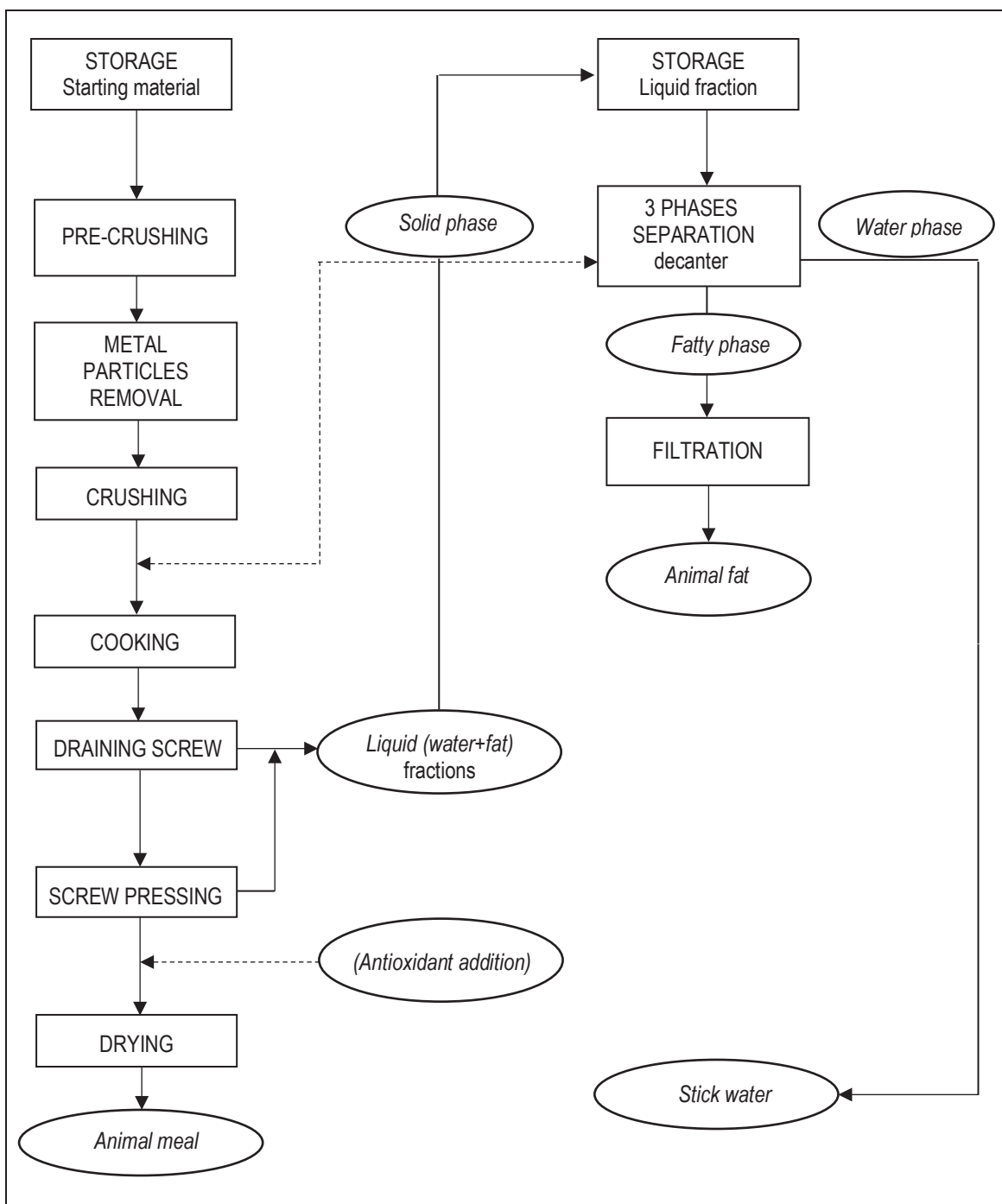


Figure 2 - Flow sheet of wet rendering process (original drawings from Lipitalia 2000 SpA)

animal fats cannot be regarded as a suitable source of tocopherols and this is confirmed by the very low concentration found in analysed samples. Looking at the fatty acids composition of samples we can see that nearly the half part of fats is constituted of saturated fatty acids, mainly palmitic and stearic acid, monounsaturated fatty acids (namely oleic acid and some palmitoleic acid) are the second constituent in terms of mass, n-3 polyunsaturated are poorly represented, while n-6 polyunsaturated fatty acids are commonly in the range of 10%.

## UNWANTED CONSTITUENTS

Animal fats for non-food use, being prepared from waste material contain several different unwanted products creating some problem during their use as a fuel as they are or in the biodiesel form. The main problems are coming from plastic polymeric material, such as polyethylene, polyamide and others. These materials, nothing having to do with fatty materials enter in the production chain in form of plastic bags where waste material is collected before rendering

**Table II - Processing methods (from Regulation (UE) 142/2011)**

**Processing method 1 (pressure sterilization)**

*Particle size reduction*

If the particle size of the animal by-products to be processed is more than 50 millimeters, the animal by-products must be reduced in size using appropriate equipment, set so that the particle size after reduction is no greater than 50 millimeters. The effectiveness of the equipment must be checked daily and its condition recorded. If checks disclose the existence of particles larger than 50 millimeters, the process must be stopped and repairs made before the process is resumed.

*Time, temperature and pressure*

The animal by-products with the particle size of no greater than 50 millimeters must be heated to a core temperature of more than 133°C for at least 20 minutes without interruption at a pressure (absolute) of at least 3 bars. The pressure must be produced by the evacuation of all air in the sterilization chamber and the replacement of the air by steam ('saturated steam'); the heat treatment may be applied as the sole process or as a pre- or post-process sterilization phase

The processing may be carried out in batch or continuous systems.

**Processing method 2**

*Particle size reduction*

If the particle size of the animal by-products to be processed is more than 150 millimeters, the animal by-products must be reduced in size using appropriate equipment, set so that the particle size after reduction is no greater than 150 millimeters. The effectiveness of the equipment must be checked daily and its condition recorded. If checks disclose the existence of particles larger than 150 millimeters, the process must be stopped and repairs made before the process is resumed.

*Time, temperature and pressure*

After reduction the animal by-products must be heated in a manner which ensures that a core temperature greater than 100°C is achieved for at least 125 minutes, a core temperature greater than 110°C is achieved for at least 120 minutes and a core temperature greater than 120°C is achieved for at least 50 minutes. The core temperatures may be achieved consecutively or through a coincidental combination of the time periods indicated.

The processing must be carried out in a batch system.

**Processing method 3**

*Particle size reduction*

If the particle size of the animal by-products to be processed is more than 30 millimeters, the animal by-products must be reduced in size using appropriate equipment, set so that the particle size after reduction is no greater than 30 millimeters. The effectiveness of the equipment must be checked daily and its condition recorded. If checks disclose the existence of particles larger than 30 millimeters, the process must be stopped and repairs made before the process is resumed.

*Time, temperature and pressure*

After reduction the animal by-products must be heated in a manner which ensures that a core temperature greater than 100°C is achieved for at least 95 minutes, a core temperature greater than 110°C is achieved for at least 55 minutes and a core temperature greater than 120°C is achieved for at least 13 minutes. The core temperatures may be achieved consecutively or through a coincidental combination of the time periods indicated.

The processing may be carried out in batch or continuous systems.

**Processing method 4**

*Particle size reduction*

If the particle size of the animal by-products to be processed is more than 30 millimeters, the animal by-products must be reduced in size using appropriate equipment, set so that the particle size after reduction is no greater than 30 millimeters. The effectiveness of the equipment must be checked daily and its condition recorded. If checks disclose the existence of particles larger than 30 millimeters, the process must be stopped and repairs made before the process is resumed.

*Time, temperature and pressure*

After reduction the animal by-products must be placed in a vessel with added fat and heated in a manner which ensures that a core temperature greater than 100°C is achieved for at least 16 minutes, a core temperature greater than 110°C is achieved for at least 13 minutes, a core temperature greater than 120°C is achieved for at least eight minutes and a core temperature greater than 130°C is achieved for at least three minutes. The core temperatures may be achieved consecutively or through a coincidental combination of the time periods indicated.

The processing may be carried out in batch or continuous systems.

or as nylon food containers in the case of expired food products sent to rendering for fat and protein recovery. Obviously, the problem is not related to big pieces that can be easily removed during the feed-

**Processing method 5***Particle size reduction*

If the particle size of the animal by-products to be processed is more than 20 millimeters, the animal by-products must be reduced in size using appropriate equipment, set so that the particle size after reduction is no greater than 20 millimeters. The effectiveness of the equipment must be checked daily and its condition recorded. If checks disclose the existence of particles larger than 20 millimeters, the process must be stopped and repairs made before the process is resumed.

*Time, temperature and pressure*

After reduction the animal by-products must be heated until they coagulate and then pressed so that fat and water are removed from the proteinaceous material. The proteinaceous material must then be heated in a manner which ensures that a core temperature greater than 80°C is achieved for at least 120 minutes and a core temperature greater than 100°C is achieved for at least 60 minutes. The core temperatures may be achieved consecutively or through a coincidental combination of the time periods indicated.

The processing may be carried out in batch or continuous systems.

**Processing method 6** (for Category 3 animal by-products originating from aquatic animal or aquatic invertebrates only)*Particle size reduction*

1. The animal by-products must be reduced to a particle size which is no greater than: (a) 50 mm, in case of heat treatment in accordance with point 2(a); or (b) 30 mm, in case of heat treatment in accordance with point 2(b). They must then be mixed with formic acid to reduce and maintain the pH to 4,0 or lower. The mixture must be stored for at least 24 hours pending further treatment.

*Time, temperature and pressure*

After reduction, the mixture must be heated to: (a) a core temperature of at least 90°C for at least 60 minutes; or (b) a core temperature of at least 70°C for at least 60 minutes. When using a continuous flow system, the progression of the product through the heat converter must be controlled by means of mechanical commands limiting its displacement in such way that at the end of the heat treatment operation the product has undergone a cycle which is sufficient in both time and temperature.

The processing may be carried out in batch or continuous systems.

**Processing method 7**

Any processing method authorized by the competent authority where the following have been demonstrated by the operator to that authority: (a) the identification of relevant hazards in the starting material, in view of the origin of the material, and of the potential risks in view of the animal health status of the Member State or the area or zone where the method is to be used; (b) the capacity of the processing method to reduce those hazards to a level which does not pose any significant risks to public and animal health; (c) the sampling of the final product on a daily basis over a period of 30 production days in compliance with the following microbiological standards:

(i) Samples of material taken directly after the treatment: *Clostridium perfringens* absent in 1 g of the products

(ii) Samples of material taken during or upon withdrawal from storage: *Salmonella*: absence in 25g;  $n = 5$ ,  $c = 0$ ,  $m = 0$ ,  $M = 0$ ; *Enterobacteriaceae*:  $n = 5$ ,  $c = 2$ ;  $m = 10$ ;  $M = 300$  in 1 g

where:

$n$  = number of samples to be tested;

$m$  = threshold value for the number of bacteria; the result is considered satisfactory if the number of bacteria in all samples does not exceed  $m$ ;

$M$  = maximum value for the number of bacteria; the result is considered unsatisfactory if the number of bacteria in one or more samples is  $M$  or more; and  $c$  = number of samples the bacterial count of which may be between  $m$  and  $M$ , the samples still being considered acceptable if the bacterial count of the other samples is  $m$  or less.

stock selection step. The problem comes from the small pieces sent to the cooking process in which they melt and become soluble/finely dispersed in the rendered fat. It is common to receive crude animal fats containing 300-500 mg/kg of polyethylene. If this fat is used without removal of polyethylene the engine filters will result blocked by this polymer and the engine will easily stop. The problem of polymers removal is very difficult to approach, and literature does not help with it. A recent research using one of most effective databases did not return any result. For this reason, animal fats processors have developed some "homemade" procedures for this purpose. In fact, it

is well known that acid degumming, chemical refining and bleaching earth treatments are useful to reduce the polyethylene content of animal fats. On the other hand, both deodorisation and physical refining do not have any effect on this contamination. The refining process in general allows to reduce the plastic contamination down to 50 mg/kg or less.

Looking at the analytical method ISO 6656:2012 'Determination of polyethylene type polymers' [12], an acidic pre-treatment of the sample using a mixture of 10% concentrated HCl and 90% ethanol is contemplated to split any possible presence of soaps, but the strength of the treatment could act also in some

**Table III** - Chemical parameters found in a 36 samples analysis of animal fats in Europe (from Nuchi *et al.* [10])

Parameter	Unit	Min. Value	Max. Value	Median Value	Average Value
Moisture	%	0	0.70	0.08	0.14
Acid Value	mg KOH/g	0	47.5	6.6	8.7
Monoglycerides	%	0	1.4	0	0.12
Diglycerides	%	0	13.5	2.1	2.7
Peroxide Value	meq O <sub>2</sub> /kg	0	6.8	1.8	2.2
p-anisidine Value	--	0.1	13.4	3.2	3.8
Polymer content	%	0.1	1.1	0.1	0.2
Total tocopherols	mg/kg	0.1	32.2	3.4	6.1
Total tocotrienols	mg/kg	0	50.5	0.8	7.6
Saturated Fatty Acids	%	27.1	59.7	44.1	41.8
Monounsaturated Fatty Acids	%	38.0	55.6	45.7	45.6
n-3 Polyunsaturated Fatty Acids	%	0.3	3.0	0.7	1.1
n-6 Polyunsaturated Fatty Acids	%	1.8	28.7	8.9	11.5

way to make polyethylene available for isolation, like during the acid degumming step or in contact with (acid) activated bleaching earths. An additional indirect confirmation comes from the fact that filter aids are not as effective as the activated bleaching earths in polyethylene removal.

The detection of plastic contamination in a blocked filter is quite easy to carry out if the solid material is carefully deprived from the fat content by means of subsequent washings with hexane [12]. The solid purified material is then analysed by means of FT-IR and the obtained spectrum compared with the one of reference materials.

## THE PROBLEM OF IMPURITIES

By definition, impurities are intended as the solid material that remains non-soluble in analytical solvent (generally light petroleum) and can be isolated by filtration and weighed. In other terms the evaluation of impurities has the meaning of quantifying the non-fat material and it consists of rust, dust, earth, fragments of bones, proteins, etc.

The original meaning of this parameter was strongly linked to the processing losses, but when fats are used for energy production, the amount of insoluble material becomes a critical point for operation. In fact, during our all-day experiences we received several samples isolated from blocked filters that stopped the engine operation.

The removal of solid contaminants seems to be a very easy operation, but in some cases, the amount of solid material, as well as the high viscosity and melting point of the feedstock may create practical problems. A possibility to overcome these problems lies in the coupling of acid degumming and filtration, that allows to remove, *in toto* or in part, phosphorous, metal contamination and solid particles in particular if gum separation is carried out by means of a centrifuge.

## THE PROBLEM OF PHOSPHOROUS

Phosphorous in non-edible animal fats can come from different sources, that is to say from phospholipids, sphingolipids as lipid material, but also in the inorganic form if bones have been used as a feedstock for rendering. In this case, Calcium phosphate can be found even in huge amounts. The contemporary presence of Calcium (and Magnesium) and phospholipids can make these last non-hydratable and difficult to remove from the oil mass, without an acidic treatment [14]. The meaning of this operation is to split the Calcium bond between Ca and P linked in phospholipids allowing the transformation of non-hydratable phospholipids (NHP) into hydratable phospholipids (HP). Phosphorous is an unwanted contaminant in oils and fats delivered for energy purposes, because of its tendency to form insoluble precipitates under heating and in presence of water. These precipitates are in glue-like form and may create big problems in filters, pipes, pumps and nozzles. In addition, other problem due to the presence of P can arise when a denox device is installed in the exhaust post treatment after the engine. In this case, it is well known that P can poison the denox catalyst by deposition of a thin layer of P<sub>2</sub>O<sub>5</sub> acting as an isolating layer. For these reasons P concentration is regulated both in EN 14214 [15] specification for biodiesel (max. 4 mg/kg) and in pure fat/oil for energy purposes (max 15, 100 or 300 mg/kg, according to the different specification classes as in UNI 11163 Standard) [16].

P removal is a well-known technology and it is generally obtained by mixing the crude fat with a 1-2% aqueous solution of ortho-phosphoric acid or citric acid (at approx. 10 % concentration in acid). The correct amount of acid to be used can be calculated starting from the knowledge of the concentration of Calcium and Magnesium. The stoichiometric amount of acid calculated in this way is generally increased 4 to 10 times in order to achieve an efficient P removal.



al [14]. For high acidity samples that are not chemically refined, after the separation of the precipitate, a washing with a 1% citric acid solution to remove traces of chemicals used and to avoid emulsions could be necessary. For some samples the removal of P can be very difficult and sometimes the standard treatment is not effective to achieve the necessary reduction. In this case some help can come from the treatment with synthetic silica and bleaching earths. The intimate contact obtained by means of very effective static or dynamic mixers is one of the key facts for the success of the operation.

## THE PROBLEM OF SULPHUR

Also, the presence of Sulphur can create several problems in the use and in the preparation of biofuels from animal fats. The already mentioned biodiesel European specification EN 14214 [15] reports a maximum S content of 10 mg/kg, in line with classic diesel fuels, while for native fats used for energy purposes up to 10, 200 or 500 mg/kg can be tolerated, according to the already mentioned Standard [16]. The concentration of S in fuels must be reduced to prevent the phenomenon of acid rains and a strong campaign of S reduction started many years ago worldwide. Describing the chemical structure of molecules containing S in oils and fats, in particular in animal fats, is very difficult. While an important literature can be found in S containing product from rapeseed oil [17], the knowledge for animal fats is nearly zero. What we can say is that S can be present in animal fats in several different structures and, if we exclude the presence of S containing aminoacids such as cystine, cysteine and methionine in proteins that can be efficiently removed by filtration, other sources of S are surely present in fats. The Lipid Handbook [18], a reference text for lipid chemistry, only contains some small citation about Sulphatites, defined as 3-sulphate esters of galactosylcerebrosides, found in mammalian tissues or sulphated glucosylglycerides (only one example in the vegetable kingdom), alkyl sulphates in some microorganisms or chlorosulpholipids in fungi and algae.

Sulphur in animal fats is not only present in one family of molecules, but it is spread within different classes. An indirect evidence of this fact comes from a series of tests carried out in our lab in studying the fate of Sulphur during a complete transformation chain of an animal fat. In Table IV and V the synthesis of these experiences carried out on two different animal fats of different origin (Sample 1 and Sample 2) is reported. Also, the possibility of S addition at the double bonds of fatty acids cannot be excluded if in some parts of the process sulphuric acid is used [19].

From the observation of data reported in Tables IV and V we can remark that every unit operation

removes some fraction of S containing molecules, but the only notable effect in reduction can be obtained in case of chemical refining and in case of biodiesel distillation. Using classic technologies, the complete S removal remains a dream.

Another experience carried out on a third Cat. 3 sample containing 44 mg/kg of S allowed to reduce the

**Table IV - Sulphur variation during a complete chemical refining chain and biodiesel production - Animal fat Cat. 3, Sample 1**

Sample	S content mg/kg	Reduction from initial concentration (%)
Crude animal fat	79	0
Degumming with H <sub>3</sub> PO <sub>4</sub>	71	10
Chemical refining	40	49
Bleaching TDA + silica	44	44
Deodorized 2 hours × 230°C × 1 mbar	37	53
Biodiesel from refined fat	40	49
Biodiesel after distillation	25	68

**Table V - Sulphur variation during a complete chemical refining chain and biodiesel production - Animal fat Cat. 1, Sample 2**

Sample	S content mg/kg	Reduction from initial concentration (%)
Crude animal fat	79	0
Degumming with H <sub>3</sub> PO <sub>4</sub>	65	18
Bleaching TDA + silica	59	25
Physical refining 7 hours × 260°C × 1 mbar	55	30
Biodiesel from refined fat	48	39
Biodiesel after distillation	26	67

content down to 38 mg/kg after bleaching (14% reduction) and to 26 mg/kg (41% reduction) after physical refining.

From these data and other experiences made during these years we can conclude that:

- S is present in animal fats in different forms
- Some unit operations such as chemical refining, (physical refining) and biodiesel distillation allow a partial reduction of S content
- Looking at the different behaviour of different animal fat samples, the ratio between the different S containing molecules may have huge variations.

## THE INORGANIC CONTAMINATION

Depending on the quality and on the origin of the starting material the rendered fat can contain even important amounts of inorganic contaminants. Calcium and Magnesium and in lesser concentration Sodium and Potassium derive from the feedstock, while

Iron, Aluminium, Zinc can come from the equipment. In every case these contaminants must be removed, and this is possible by means of the acidic treatment during degumming. In fact, removing inorganic contamination is not only a matter of physical removal, because in several cases metals are present as fatty acid soaps and these must be split to free fatty acids for an effective removal. Acidic treatment can be carried out by means of aqueous solutions of ortho phosphoric acid or citric acid. This last option must be preferred in case of high inorganic contamination, because apart its acidic property it also can act as a sequestrant, allowing the transfer of metal ions in the aqueous layer. When processing high acidity animal fats, a second washing using diluted acid can be useful to complete the operation and avoid emulsions in case only water is used.

## CONCLUSIONS

This paper represents an overview of the experience achieved during our professional activity in the sector of non-food animal fats, considering also what has been published on this issue. This sector has evolved greatly from the beginning of the new century, after many years of relative quietness, when non-food animal fats were mainly used for the preparation of glycerol and fatty acids from splitting. The driving force for these changes was double:

- At first the onset of Mad Cow Disease, causing the Bovine Spongiform Encephalopathy and the relative European Regulation with the classification of fats in three different categories having different risk hazards. The plants had to be adapted to this new Regulation and after some years, when the problem declined, and Category 1 animal fats were authorised to be delivered instead of destroyed within the production unit. In this case a treatment for sterilisation before delivery became mandatory.
- Secondly, when companies producing biodiesel and electric power using diesel static engines began looking for cheaper and more sustainable materials to substitute vegetable oils and fats, the demand of animal fats raised enormously, with the double effect of creating tension on the selling prices and research and recovery of every material that could be classified as fat. This resulted in the arrival of very bad quality fats that need to be strongly processed before use, considering that the specification for the final product does not change.

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