



PERCAL PROJECT

Conversion routes of the Organic Fraction of Municipal Solid Waste into high added value products

Bio based Industries

Consortium

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1. Introduction

This guide illustrates the PERCAL project technologies and provides guidance on the safety, regulatory, economic and environmental aspects of the intermediate bioproducts developed during the project.

1.1 PERCAL project aim and objectives

In 2018, the EU-28 produced more than 250 million Tonnes of Municipal solid waste, which means an average of 489kg/capita per year (Source: Eurostat). 30-50% of the MSW is organic waste. The current treatments of the MSW are landfilling (23%), recycling (31%), incineration disposal and energy recovery (29%) and composting and digestion (17%)

The amount of MSW generation is increasing at an annual rate of 10%. In spite of the strong shift from MSW landfilling to recycling of materials such as plastics, wood or metals and energy production from the organic fraction (mainly by incineration), there is a challenge to turn the biogenic fraction into higher value products other than or besides bioenergy.



Cost competitive value chains producing added-value products from heterogeneous and complex waste material create solutions for the environmental problem of increasing waste flows, reducing pressure on virgin resources, and increasing the competitiveness of European industry. Nowadays there a several initiatives funded at national and EU level developing innovative solutions to valorise waste. The Bio-based Industries Joint Undertaking (BBI JU) has focused all its efforts to fund projects in the bio-based sector, and most of them addressing the development of technologies and biorefineries able to tackle the waste problem.

PERCAL project, funded by the BBI JU, presents an opportunity to exploit Municipal Solid Waste (MSW) as a feedstock to develop intermediate chemical products at high yield and low impurity level with huge industrial interest. PERCAL combines several technologies and processes starting from different Technology Readiness Levels (TRL) to valorise the organic fraction of MSW in bio-based chemical products.

PERCAL's starts with the collection of the OFMSW to be treated at PERSEO Biorefinery Plant. The main process of PERSEO Biorefinery is the obtention of second-generation bioethanol. PERSEO Biorefinery is based in a biotechnological patented technology PERSEO Bopethanol® developed by IMECAL jointly with CIEMAT. The plant can process up to 25 t/day of organic fraction of MSW. PERCAL used this knowledge/background as a starting point to produce new high added value products from MSW. PERCAL addresses a cascade valorisation of the MSW which components, are complementary to the bioethanol production. The bioproducts and technologies developed may be integrated into current waste management facilities:



- Lactic acid (LA) to produce: 1) Eco-friendly ethyl lactate solvents by reactive distillation from lactic acid & bio-ethanol to be used in cleaning products and inks and 2) hot-melt adhesives for cardboard and other non-food applications by reactive extrusion.
- Succinic acid (SA) as an intermediate building blocks to production of polyols for the polyurethane industry.
- Biosurfactants by chemical and/or microbiological modification of protein and lipid fraction from remaining fraction of MSW fermentation.

1.2Waste as a feedstock

Municipal waste consists of waste collected by or on behalf of municipal authorities and disposed of through waste management systems. Municipal waste consists mainly of waste generated by households, although it also includes similar waste from sources such as shops,

restaurants, offices and public institutions.¹ PERCAL project target feedstock includes the biodegradable fraction of this waste which can come from two different origins:

- **4** sorted organics from municipal bring points or kerbside collection (separate collection)
- organics remaining after processing in mechanical biological treatment (MBT) plants or similar centralised sorting (non-separate collection - with higher inert materials content).

This organic fraction of municipal solid waste (OFMSW), also referred as biowaste, is mainly composed of two main streams: food waste from kitchens / households and green waste from gardens. Chemically, the OFMSW contains mainly carbohydrates (cellulose, hemicellulose, starch, pectins), proteins and lipids, which are all useful raw material that can be converted into biobased products. The valorisation of this fraction not only helps to solve environmental pollution, but also contributes to the transition from a linear to a renewable circular economy. The conversion of this OFMSW into new biobased chemical intermediates (bioethanol, lactic acid, succinic acid) and final products (biosurfactants, adhesives, solvents, polyester-polyols) is the aim of PERCAL project.

In the MSW stream, waste is broadly classified into organic (biowaste) and inorganic. Usually, waste composition is categorized as organic, paper, plastic, glass, metals, and 'other.' Waste composition is influenced by several factors. As a country urbanizes and populations become wealthier, consumption of inorganic materials (such as plastics, paper, and aluminum) increases, while the relative organic fraction decreases. Generally, low and middle-income countries have a high percentage of organic matter in the urban waste stream, ranging from 40 to 85% of the total. Paper, plastic, glass, and metal fractions increase in the waste stream of middle- and high-income countries².

Regarding MSW generation, each person generates, on average, 475 kg of MSW every year in Europe. Since around 40-50% of this waste corresponds to bio-waste, it can be estimated that around 100 million tons of biodegradable waste derived from MSW are yearly produced in the EU. Municipal biowaste is therefore considered a very interesting feedstock for its valorization in future biorefineries due to its abundance, low cost and high potential value.

1.3 Regulations and policies

In the recent years, more and more efforts are being made from different research organizations, governments, industries and civil society to move towards a more sustainable bioeconomy, trying to reduce waste, pollutants and globally speaking, reducing our dependence on fossil-based resources. Although this worldwide movement is getting stronger over the time, a well-established regulatory framework which allows the implementation of biobased technologies and the go-to-market for bio-based products is still in preliminary stages.

¹ <u>http://ec.europa.eu/eurostat/statistics-explained/index.php/Glossary:Municipal_waste</u>

² What a waste. A Global Review of Solid Waste Management, World Bank, 2012

For many products and applications there are still regulatory gaps which limit their use in specific fields, as it is the case for bio-chemicals coming from waste.

From the European Commission this matter has been tackled through the European Green Deal which includes a plan and a series of actions addressed to boost a circular economy in Europe. This global strategy includes the development of an appropriate and global regulatory framework.

1.3.1 The European Green Deal and the European Circular Economy Action Plan

The European Green Deal³ is the European Commission's plan to make the EU's economy sustainable thorugh an action plan to boost the efficient use of resources by moving to a clean, circular economy; and to restore biodiversity and cut pollution. The objective is that the EU is climate neutral by 2050. For that, The European Commission has adopted a new Circular Economy Action Plan.

The publication of the preliminary European Circular Economy Package⁴ by the EU Commission in December 2015 paved the way for a resource-efficient society and sustainable recycling industry across Europe. The new action plan announced initiatives along the entire life cycle of products, targeting the design, promoting circular economy processes, fostering sustainable consumption, and aiming to ensure that the resources used are kept in the EU economy for as long as possible. After three year after adoption, the final report of the European Circular Economy action plan was published in March 2019. In addition to setting out an action plan, the preliminary report also recommended revisions to key EU waste legislation, including the Landfill Directive (1999/31/EC) and the Waste Framework Directive, with the aim of avoiding, reusing and recycling more waste in the future. In particular, the amendments to waste management legislations included the following targets⁵:

- A common EU target for recycling 65% of municipal waste by 2035;
- A common EU target for recycling 70% of packaging waste by 2030;
 - There are also recycling targets for specific packaging materials: Paper and cardboard (85%), ferrous metals (80%), aluminium (60%), glass (75%), plastic (55%) and wood (30%);
- A binding landfill target to reduce landfill to maximum of 10% of municipal waste by 2035;
- Separate collection obligations are strengthened and extended to hazardous household waste (by end 2022), bio-waste (by end 2023), textiles (by end 2025).
- Minimum requirements are established for extended producer responsibility schemes to improve their governance and cost efficiency.

³ <u>https://ec.europa.eu/info/strategy/priorities-2019-2024/european-green-deal_en</u>

⁴ <u>https://ec.europa.eu/environment/circular-economy/</u>

⁵ https://ec.europa.eu/environment/circular-economy/first_circular_economy_action_plan.html

Prevention objectives are significantly reinforced, in particular, requiring Member States to take specific measures to tackle food waste and marine litter as a contribution to achieve EU commitments to the UN SDGs.

As bio-waste is the largest fraction of Europe's municipal waste stream (comprising, between 40-50% by weight),⁶ the 10% landfill target can only be met through sustainable bio-waste management, including traditional composting and anaerobic digestion processes but also new complementary technologies such as the ones proposed in PERCAL project.

1.3.2 Waste Directive

Directive 2008/98/EC on waste (Waste Framework Directive), and Directive (EU) 2018/851 (amendment of Directive 2008/98/EC on waste), sets the basic concepts and definitions related to waste management, such as definitions of waste, recycling, recovery. It explains when waste ceases to be waste and becomes a secondary raw material, and how to distinguish between waste and by-products.

The Directive sets some the waste management principles: it requires that waste must be managed without endangering human health and harming the environment, and in particular without risk to water, air, soil, plants or animals, without causing a nuisance through noise or odours, and without adversely affecting the countryside or places of special interest. Waste legislation and policy of the EU Member States shall apply as a priority order the following waste management hierarchy:



Figure 1. Waste management hierarchy.

The amendment Directive aims to prevent waste and increase the recycling of municipal waste, including the subsequent phasing out of landfilling practices. This is done by strengthening the waste hierarchy, in a way in which each State Member is encouraged to take specific measures to prioritize prevention, re-use and recycling as opposed to landfilling and incineration. In this sense, the Directive sets new targets for the preparation for re-use and recycling of municipal solid waste (MSW); 55% by 2025, 60% by 2030 and 65% by 2035; and

⁶ Managing Municipal Solid Waste, EEA Report No2/2013

it also sets that the maximum quantity (10%) of municipal waste landfilled by 2035. Furthermore, the Directive sets rules relatives to the objectives calculations and for example establish that the MSW eliminated by incineration and the waste produced in operations of stabilization of the biodegradable fraction of MSW to be landfilled will be considered as landfilled.

Among different measures, the separate collection of bio-waste (biodegradable garden and park waste, food and kitchen waste from households, restaurants, caterers and retail premises and comparable waste from food processing plants), not only for composting and digestion, but also for the use of environmentally safe materials produced from bio-waste are encouraged by the directive. This will be mandatory for bio-waste by 2023 and the same will for textile waste by 2025.

1.3.3 REACH compliance

REACH is a Regulation of the European Union, adopted to improve the protection of human health and the environment from the risks that can be posed by chemicals, while enhancing the competitiveness of the EU chemicals industry. In principle, REACH applies to all chemical substances. Therefore, the regulation has an impact on most companies across the EU. REACH places the burden of proof on companies. To comply with the regulation, companies must identify and manage the risks linked to the substances they manufacture and market in the EU. They have to demonstrate how the substance can be safely used, and they must communicate the risk management measures to the users.

REACH is a complex Regulation; therefore, some concepts must be very clear in order to their correct accomplishment. In this sense, according to REACH regulation, polymers (as polylactic acid hotmelts, polyols and polyurethanes) are exempted from registration obligations nowadays for themselves, however as it is described in Article 6(3) of REACH, any manufacturer or importer of a polymer shall submit a registration to the Agency for the monomer substance(s) or any other substance(s), that have not already been registered by an actor up the supply chain, if both the following conditions are met:

- the polymer consists of 2 % weight by weight (w/w) or more of such monomer substance(s) or other substance(s) in the form of monomeric units and chemically bound substance(s)
- the total quantity of such monomer substance(s) or other substance(s) makes up one tonne or more per year.

To develop PERCAL project different reactants consisting of substances and mixtures have been used which previously has been registered.

Regarding the developed products (intermediates, cascade and final products) according to REACH, some of them are considered mixtures and others are classified as substances. Thus, bioethanol, lactic acid, succinic acid, ethyl lactate and lactide are included in the last group, they are substances and, in this sense, to commercialize them it will be required its previous registration. The rest of products obtained are considered mixtures: biosurfactants, window cleaners and printing inks, hotmelts and polyols for polyurethanes (PUR).

On the other hand, the reactants used in this project are considered mixtures and substances, so we have taken into account in their selection that the substances included in these mixtures and the substances itself have been previously registered by their suppliers.

1.3.3.1 REACH for recovered substances

REACH also applies to recovered substances and an explanation of these application is described in detail in the "Guidance on waste and recovered substances" (<u>https://echa.europa.eu/documents/10162/23036412/waste recovered en.pdf/657a2803-710c-472b-8922-f5c94642f836</u>). Article 2(2) of REACH provides that "waste as defined in Directive 2006/12/EC4 of the European Parliament and of the Council is not a substance, preparation or article within the meaning of Article 3 of this Regulation." Therefore, REACH requirements for substances, mixtures and articles do not apply to waste.

On the other hand, as soon as a material 'ceases to be waste', REACH requirements apply in principle in the same way as to any other material, with a number of exceptions granted conditionally. The point at which waste 'ceases to be waste' has been the subject of long debates. According to Article 6 (1) and (2) of the new Waste Framework Directive, certain specified waste shall cease to be waste when it has undergone a recovery operation and complies with specific criteria to be developed in line with certain legal conditions, in particular: (a) the substance or object is commonly used for specific purposes; (b) a market or demand exists for such a substance or object; (c) the substance or object fulfils the technical requirements for the specific purposes and meets the existing legislation and standards applicable to products; and (d) the use of the substance or object will not lead to overall adverse environmental or human health impacts.

Thus, in the same way as any other substance falling under the scope of REACH, recovered substances are, in principle, subject to REACH registration requirements. The legal entity performing the final recovery

should check whether the recovered substance is exempt from registration because it is listed in Annex IV or covered by Annex V of REACH. On the other hand, article 2(7)(d) of REACH provides an exemption for recovered substances under certain conditions:

- 4 (d) Substances, on their own, in mixtures or in articles, which have been registered in accordance with Title II and which are recovered in the Community if: (i) the substance that results from the recovery process is the same as the substance that has been registered in accordance with Title II; and; (ii) the information required by Articles 31 or 32 relating to the substance that has been registered in accordance with Title II is available to the establishment undertaking the recovery."

In PERCAL, the substances obtained from the waste are not included in this exemption, so they are subjected to registration as any other substance.

1.3.3.2 MSDS Compliance

To accomplish REACH Regulation and analyze the potential risks of the substances and mixtures used along the project, the MSDS (material safety data sheet) is the most suitable tool. Obviousy, before entering to the market, the corresponding MSDS for the developed products must be prepared too if applies. Safety data sheets (SDS) have been a well-accepted and effective method for the provision of information to recipients of substances and mixtures in the EU. The SDS provides a mechanism for transmitting appropriate safety information on substances and mixtures where:

- a substance or a mixture meets the criteria for classification as hazardous according to CLP; or,
- a substance is persistent, bioaccumulative and toxic (PBT) or very persistent and very bioaccumulative (vPvB), according to the criteria given in Annex XIII of REACH, or;
- a substance is included in the candidate list for eventual authorization according to Article 59 (1) of REACH for any other reasons (see Article 31(1) of REACH).

Under certain conditions some mixtures which do not meet the criteria for classification as hazardous according to CLP also require an SDS to be prepared or be made available on request (see Article 31(3) of REACH and notes to tables 3.4.6, 3.6.2, 3.7.2, 3.8.3 and 3.9.4 of Annex I of CLP).

In the EEA, the required SDS format and content are defined by Article 31 and Annex II of REACH. These have been adapted to align them with the GHS requirements, in particular with the "guidance on the preparation of safety data sheets (SDS)" given in Annex 4 of the GHS as well as to be fully in line with the CLP Regulation. This version of the Guidance on the compilation of SDSs reflects text of the revision of Annex II of REACH as replaced by the Annex to Regulation (EU) 2015/830 (amending REACH) with effect from 1 June 2015.



until May 31, 2017 without having to comply with the annex to this Regulation

Figure 2. MSDS Regulatory adaptations along the last years

2. PERCAL Technologies

2.1 PERSEO Bioethanol® Process & Technology

PERSEO Bioethanol® is an innovative technology to transform organic residue (biodegradable municipal solid waste, HORECA sector, paper and cardboard) into high value commercial products: 2G bioethanol, to be used as liquid fuel or as a chemical building block for the chemical industry, and an organic residue with high calorific value for the generation of heat and electricity via cogeneration or biomethane biofertilizer and/or via anaerobic digestion/compost. PERSEO technology can be adapted for the production of other high value bio-products by customizing the

pre-treatment step in order to be applied to other fermentative processes.

PERSEO Bioethanol® is a patented biotechnology fully compatible with current waste treatment plants. The process can be adapted as an intermediate section of increased value in current plants, including incineration, anaerobic digestion or composting. It can enhance the waste treatment value chain and improve economic and environmental figures of waste management.

KEY ADVANTAGES

Non-valuable organic waste as a feedstock Patented process and technology, demonstrated at a semi-indutrial scale (TRL7-8), guaranteeing a stable and high yielding process Potential for valorisation of all currents and streams into bio-products Integrated biorefinery, fully compatible with current waste management plants Alligned with the EU circular economy strategy

The first step in the chain to convert waste into valuable bioproducts

Designed as a flexible process technology, it allows the valorisation of all the process streams into market valuable products. In PERCAL, the valorisation routes start from two different steps in the process. At the beginning, the organic waste is hydrolysed using specific enzyme cocktails. This hydrolysis facilitates the release of the simple and complex sugars from the organic residues. The liquid fraction of the hydrolysate is rich in sugars which can be further fermented to obtain Lactic acid (LA) and Succinic acid (LA).

In subsequent step of the PERSEO bioethanol® process, after the simultaneous saccharification and





2.2 Reactive extrusion

PERCAL is exploiting the Organic Fraction of Municipal Solid Waste (OMSW) as feedstock to obtain first lactic acid (LA) as chemical intermediate for the production of lactide. In PERCAL, the lactide is produced in a three-step process. In the first step, a low molecular weight PLA oligomer is obtained by polycondensation of LA with simultaneous water distillation. In a second step, the oligomer is depolymerized in presence of catalyst by a mechanism that involves a back-biting reaction of the PLA and dimerization to lactide, that is distilled to yield crude lactide. Finally, the crude lactide is recovered via recrystallization and drying.

The obtained lactide is the main raw material in the synthesis hot melt adhesives (HMAs). PLA copolymers are obtained by ring opening polymerization (ROP) of lactide in presence of other co-monomers, initiators and catalyst by means of Reactive Extrusion process (REX). The obtained copolymer is finally compounded with natural tackifiers and additives in a twinscrew extruder and/or grafted with maleic

anhidryde by REX to obtain the final biobased hot melt adhesives (HMAs).



PERCAL bio-HMAs were validated by Lap shear test (according to ISO 4587) in different carboard boxes, showing or improved performance in comparison with reference fossil-based EVA hot melt adhesives used for closing of cardboard boxes in the packaging sector.

Reactive Extrusion was the core technology to synthesize and modify different PLAs and their copolymers from PERCAL lactide.

KEY ADVANTAGES

Highly versatile continuous polymerization reactor Ability to handle high viscosity system Almost free solvent process Reduced residence time compared with standard batch systems Indicated for low volume and tailor made formulations Process demonstrated at the pilot scale scale (TRL5)







2.3 Electrodialysis

ATB has developed a process in which the organic fraction of municipal solid wastes (OFMSW) are exploited for the generation of lactic acid (LA). LA is a versatile chemical, which finds applications in many fields such as pharmacy, food and chemical industry.

The process utilizes the highly specialized Bacillus coagulans for bacteria the fermentation of OFMSW hydrolysates. The microorganism is able to LA homofermentatively transform sugars into, in other words without the generation of byproducts. As a result, yields for the conversion of sugars are above 80 %. By the end of the fermentation, the broth, containing typically more than 50 g/L of LA

enters the downstream and purification process.

In electrodialysis (ED), ions are transported from one solution to another utilizing ionexchange membranes and by applying an electrical potential difference. Unlike the commercial method for separation of LA, which generates large amounts of waste the separation based (gypsum), on electrodialysis generates practically zero waste. Additionally, ED offers great flexibility in scale of production, high selectivity and levels of purification. Furthermore, it can be easily integrated to conventional fermentation processes, reducing the equipment investment cost

KEY ADVANTAGES

Highly efficient fermentation - no by-products generated
No generation of waste during downstream
Lactic acid is the intermediate in the production of polylactic acid and ethyl lactate
Process demonstrated at the pilot scale scale (TRL5)
Advantages of electrodialysis include: high selectivity and levels of purification, easy integration with conventional fermenters

Other steps such as filtrations and further ion removal by resins in exchange columns, the product enters the final step in which vacuum distillation. By the end of the process, a very clear LA solution with high purity is obtained. In PERCAL, LA is the building block in the production of PLA, a biobased polymer with mechanical properties between those of polystyrene and PET. Moreover, LA can be used in the production of the green solvent, ethyl lactate.









2.4 Membrane electrolysis

Bio-based carboxylic acids are promising platform chemicals. The conventional downstream separation and purification process employed for the recovery of these acids from fermentation broths involves the application of various unit operations increasing processing costs.

An electrochemical membrane bioreactor using an anion-exchange membrane has been developed leading to single-step separation and acidification of succinate salts into succinic acid. The novel electrochemical bioreactor enables in situ production and separation of succinic acid fermentation broth (Figure from 1). Membrane electrolysis is a process where both electrode reactions, the cathodic reduction and anodic oxidation, are linked to the transport and transfer of charged ions. The developed process involves continuous fermentation broth circulation through the cathode compartment of the electrolysis cell. The anions cross the membrane into the anodic chamber resulting in a succinic acid concentrate solution. Water reduction (cathode) occurs in the fermentation broth, generating hydroxide ions and hydrogen gas in the broth. The hydroxide ions can partly replace the sodium hydroxide (NaOH) employed for maintaining fermentation pH. At the anode, oxidation reaction takes place and the extracted ionic product is protonated to its acidic form by electrolytically generated protons.

These membranes are permeable to carboxylic acid anions such as acetic acid, lactic acid, succinic acid etc., but it is impermeable to biomass and various impurities, resulting in combined extraction, clarification, acidification and concentration steps in a single unit operation.



Figure 3. Integrated succinic acid production and separation using an electrochemical bioreactor

The integration of membrane electrolysis during fermentation coupled with in-line separation has been demonstrated at lab scale experiments using a genetically engineered *Yarrowia lipolytica* strain PSA02004 for succinic acid production (TRL 3-4). Semi-pilot scale demonstration of this novel technology will be also carried out (TRL 5).



KEY ADVANTAGES

Around 35% less NaOH required for pH control during fermentation.

Reduced unit operations for the downstream and purification process

ME technology is mainly dependent on electricity consumption that can be derived from renewable resources Environmental impact of traditional downstream separation and purification processes can be reduced by 5% CO₂ eq/kg_{SA} and 8% MJ/kg_{SA} of fossil depletion

Bio-based succinic acid for a more circular industry of polyurethanes

The membrane electrochemical bioreactor has provided integrated production and separation of succinic acid in a single step during the project. The continuous in-situ extraction of organic acids from fermentation broth improved succinic acid production efficiency. The purified succinic acid crystals from this process were employed as an intermediate to produce polyester polyols, the main components of polyurethane.



2.5 Reactive distillation

Reactive distillation is a highly efficient process that combines a chemical reaction with the simultaneous separation of products. In the PERCAL project, reactive distillation was used to produce the environmentally-friendly solvent ethyl lactate from the chemical building blocks ethanol and lactic acid.

Ethyl lactate is a green solvent that offers a number of environmental and health benefits over conventional petrol-based solvents. It is 100% bio-based, readily biodegradable and therefore, renewable in the sense of a circular economy. A main advantage of a versatile biorefinery is the economic resilience based on a broad product spectrum. By switching production the supply can be adjusted to market variations. Reactive distillation combines two chemical building blocks of the versatile biorefinery and contributes to its flexibility. The process does not require enantiomeric purity of lactic acid feedstock. Therefore, variations in quality of chemical building blocks can be compensated.

KEY ADVANTAGES

Efficient production of the green solvent ethyl lactate Feedstock entirely renewable, obtained form the biorefinery Potential to compensate quality variations of building blocks Low investment and operation costs Demonstrated at pilot scale (TRL 5)

Efficient chemical route to produce the green solvent ethyl lactate

Reactive distillation utilizes process intensification to overcome the limitation of reactant conversion caused by inhibitory effects of the side-product water. By constantly removing water from the catalytic zone the reaction is enhanced and high yield and product purity is reached in a single apparatus.

Due to process intensification the production of ethyl lactate is performed

energy efficient in compact equipment. Low operational and investment costs are expected compared to conventional production technology.

Reactive distillation was demonstrated using a glassware apparatus. After optimization actions the process is currently operable at pilot scale (TRL 5) with further scale-up potential.









2.6 NIR-based Monitoring System

Near-infrared (NIR) spectroscopy offers a rapid sample processing without the need of sample preparation. However, and despite it is a non-destructive technique, the amount of data generated by these techniques requires powerful statistics tools for the consequent processing. The development of the models associated to the technology is carried out with chemometric studies using every single product to analyze. More specifically, the product developed by IRIS to this purpose has been named after *VISUM-inline*.

The VISUM-inline is a NIR spectrophotometer, which includes a light source (tungsten halogen light bulb), sampling, detector, collimators and optical fiber. The NIR spectra are collected from two different sensors that are used in order to quantify bioethanol, succinic and lactic acid concentrations from fermentation of MSW, in the ranges 900 – 160 nm and 1000

- 25000 nm. As a simplified description *VISUM-inline* works by shooting a beam of light upon the samples for, I the aftermath, measuring their interaction and how it reflects the light radiation. Given that several parameters could affect the spectroscopy signal, IRIS has optimized different configuration setup for data acquisition, testing the feasibility of the specific targeted fermentation process in a laboratory-scale, including the monitoring of ethanol, lactic acid and succinic acid..



Figure 4. External view of the VISUM-inline monitoring system developed by IRIS and designed for the incorporation to the pipeline in PERSEO

KEY ADVANTAGES

Quick analysis with no need for sample preparation Non-destructive analysis Easy to conduct/mantain from trained staff Avoids cross-contamination of samples Good scalability of the technology from lab to industrial scale

In-line monitoring, key facts to improve the efficiency of the process:

Allows the validation of the adaptability of a versatile biotechnological plant to obtain different chemical intermediates.

Allows the unification of the monitoring and control of all key parameters in one unified biorefinery/system, thus permitting the further optimisation of the process

Opens de possibility for the in-line monitoring of the production of LA, SA, glucose and ethanol to the industrial scale; and/or any other product to be considered.

3. Safety, regulatory, economic and environmental aspects of PERCAL intermediate bioproducts

3.1 Bioethanol

3.1.1 Safety & Regulatory framework

PERCAL's bioethanol has been produced using PERSEO Bioethanol® technology from hydrolysates of the organic fraction of the MSW, and this bioethanol may be used to produce ethyl lactate together with lactic acid by reactive distillation. This bioethanol is considered as a substance under REACH Regulation and for this reason it would need its previous registration before being put into the market, as also required for the ethanol and bioethanol produced or imported in the EU. To obtain this substance only registered substances have been used, and nor SVHC or restricted substances have been used.

According to the information complied on the ECHA website (<u>https://echa.europa.eu/es/substance-information/-/substanceinfo/100.000.526</u>) ethanol is manufactured and/or imported in the European Economic Area in 1 000 000+ tonnes per year and is used by consumers, in articles, by professional workers (widespread uses), in formulation or re-packing, at industrial sites and in manufacturing. Bioethanol, currently, supposes a smaller market but anyway the biobased products are gaining day by day quote of market. Among their high amount of uses we can mention:

- Biocidal: is being reviewed for use as a biocide in the EEA and/or Switzerland, for human hygiene, disinfection, food and animals' feeds, product preservation.
- Consumer, professional and industrial uses: fuels, coating products, anti-freeze products, washing & cleaning products, biocides (e.g. disinfectants, pest control products), adhesives and sealants, inks and toners and leather treatment products, laboratory chemicals, heat transfer fluids, textile treatment products and dyes, finger paints, plant protection products, fillers, putties, plasters, modelling clay and lubricants and greases, etc.

3.1.2 Market overview

The most common global feedstocks for bioethanol are sugarcane and corn which represent 57.9% and 19.6% of the market respectively (Europe 2017 data). However, there is a growing concern over food and water shortages and the traditional agricultural feedstock for bioethanol production are no longer favoured. Alternatives to the so-called 1st generation feedstocks, in the form of lignocellulosic material, are being investigated but extraction of the fermentable sugars within this feedstock is more difficult and as yet the technology is not as matured.

Utilisation of feedstocks other than lignocellulose such as algae and municipal solid waste have also received a lot of attention in recent years.

The bioethanol market is growing and by 2025 is reported to be circa USD 64.8 billion, growing at a compound annual growth rate (CAGR) of 14% between 2020 and 2025 from USD 33.7 billion in 2020.

Ethanol as an alternative fuel for transport applications is not new and this application accounts for much of the research and innovation activities funded in couple of decades in particular. Fuels is potentially a large and an important market for bioethanol, however, within PERCAL the focus has been on the provision of ethanol primarily as an adjunct for chemical manufacture, for ethyl lactate specifically. North America is projected to be the largest contributor and take a maximum market share, in part due to the fact that the USA has a significant number of large facilities for bioethanol production. In Europe, members of ePure produced 5.81 billion litres of ethanol, of which 82% was used for fuel, 9% for industrial uses and 9% in food and beverage.

Ethanol is used for a wide range of chemicals, including commodities such as ethylene, acetic acid aldehyde, diethyl ether and ethyl acetate as well as for a range of specialty and fine chemical, including pharmaceutical and consumer care product manufacturing processes. However, the use of ethanol for higher value products such as solvents including ethyl lactate is a more attractive proposition. In addition, ethanol is used in hand sanitisers, currently a larger market than previously due to the Covid-19 global pandemic.



Figure 5. Bioethanol market, volume (%), by application, Global, 2019.

3.2 Lactic Acid (LA)

3.2.1 Safety & Regulatory framework

PERCAL Lactic acid (LA) has been produced by fermentation of hydrolysates extracted from the organic fraction of the MSW. LA has been used afterwards to produce lactide as a cascade product which is the precursor of polylactic acid (PLA). In the same way of the bioethanol, lactic acid is considered a substance in REACH, however, in this case, it must be considered as a monomer when its use is driven to the production of PLA or other polymers. It is important to highlight that in the case of employing lactic acid for the obtention of different products such as monomers and other type of products (i.e. ethyl lactate, etc.) in the same production site,

the manufacturer must register the total amount of lactic acid manufactured, specifying the amount that goes for each type of use.

In PERCAL, to obtain SA only registered substances have been applied (i.e., sodium hydroxide as pH regulator in the fermentation stage and downstream operations) and nor SVHC neither restricted substances have been used to do it.

According to the information complied on the ECHA website (<u>https://echa.europa.eu/es/substance-information/-/substanceinfo/100.000.017</u>) lactic acid is manufactured and/or imported in the European Economic Area in 100 - 1 000 tonnes per year and this substance is used by consumers, in articles, by professional workers (widespread uses), in formulation or re-packing, at industrial sites and in manufacturing. Among their high uses we can mention:

- Biocidal: is approved in the EEA and/or Switzerland for use in biocidal products more favourable for the environment, human or animal health. It is an authorised food additive.
- Consumer, professional and industrial uses: coating products, pH regulators and water treatment products, laboratory chemicals, washing & cleaning products, non-metalsurface treatment products and water treatment chemicals, it is used as an intermediate and for the manufacture of chemicals and food products, for plastics, adhesives, etc.

3.2.2 Market overview

The global lactic acid market size was valued at USD 2.64 billion in 2018 and is expected to grow at a (CAGR) of 18.7% from 2019 to 2025 resulting in an attractive potential market based on growth. Lactic acid has received significant attention in recent years due to use in the manufacture of biodegradable plastic, polylactic acid and it is usually manufactured either through chemical synthesis or fermentation. Most of the lactic acid is produced from bioderived feedstocks including corn, sugar cane and, in Europe, sugar beet. In the U.S lactic acid produced from sugarcane emerged as a dominant segment in 2018 accounting for around 40% of the total revenue share, thus suggesting that there is still scope for multi-suppliers of the material.

LA is produced through fermentation due to lower production costs and consumers awareness of the need for biobased materials. Most of the LA is produced using sugarcane, corn and cassava. Several studies suggest that a transition to from starchy materials to lignocellulosic materials (forestry waste, agricultural waste) and industrial waste as feedstock, is attractive owing to their cost-effectiveness for large-scale fermentation.

Major uses for lactic acid are shown in the chart below. PLA segment was the main application in 2018 with 28 % of the revenue share. The rise in polylactic acid production in bioplastics has seen the rapid rise of this application, together with its rising use in 3D printing filaments, textiles, and agriculture. The packaging segment is expected to be the largest owing to the unique thermal and mechanical properties offered by PLA, which makes it a suitable packaging material.

Due to their skin benefits, the increasing adoption of cosmetic products among men and transition to biobased materials, it is expected a boost to LA market in the cosmetic industry.

Pharmaceutical was the fastest-growing segment, occupying a majority of the market revenue share for over 9.1% in 2018. As noted above already, the food and drink market may not be feasible for waste derived lactic acid with the current regulatory policies in place.



Figure 6. Global lactic acid market revenue by application, 18 (%)⁷

Increasing biodegradable polymer and lactate solvents is creating new market opportunities. Co-development and/or licencing with an existing producer/supplier of lactic acid is likely to be the best option for lactic acid production; alternatively, a more attractive route could be its utilisation for ethyl lactate production with the ethanol also produced in PERCAL.

3.3 Succinic Acid (SA)

3.3.1 Safety & Regulatory framework

PERCAL Succinic acid (SA) has been produced by fermentation of hydrolysates extracted from the organic fraction of the MSW. SA has been used as precursor (monomer) for the production of polyols for the polyurethane (PU) industry. As for the lactic acid, succinic acid has to be considered as a substance and as monomer when its use is driven to the production of a polymer (polyol, polyurethane and others). To obtain succinic acid only registered substances have been applied, and SVHC or restricted substances have not been used to do it.

According to the information complied on the ECHA website (<u>https://echa.europa.eu/es/substance-information/-/substanceinfo/100.003.402</u>) succinic acid is manufactured and/or imported in the European Economic Area in 10 000 - 100 000 tonnes per year and this substance is used by consumers, in articles, by professional workers (widespread uses), in formulation or re-packing, at industrial sites and in manufacturing. Among their high uses we can mention:

⁷ www.grandviewresearch.com

Consumer, professional and industrial uses: adsorbents, fertilisers, inks and toners, washing & cleaning products, water softeners, adhesives and sealants, coating products, fillers, putties, plasters, modelling clay, perfumes and fragrances, pharmaceuticals, polymers and cosmetics and personal care products

3.3.2 Market overview

The succinic acid market size is projected to grow from USD 131.7 million in 2018 to USD 182.8 million by 2023, at a CAGR of 6.8%. The increasing demand from the industrial, personal care and food & beverage industries fuels the succinic acid market. The increasing demand from the APAC region, and the increasing adoption of succinic acid as a substitute of adipic acid in polyurethane production are the factors driving the succinic acid market. The growth of the segment is mainly attributed to the demand from the chemical industry for polybutylene succinate (PBS) and polybutylene succinate co-terephthalate (PBST), and polyurethane.

Succinic acid can be used as platform chemical for the production of 1,4-butanediol, tetrahydrofuran γ -butyrolactone as well as for the production of biopolymers such as poly(butylene succinate) and polyester polyols and is therefore a crucial intermediate. Additionally, succinic acid is a key building block of a wide range of secondary chemicals used across various applications including pharmaceutical, food & beverage, solvents & lubricants, polyurethane, and agriculture. Initial uses of bio-succinic acid were primarily as a replacement for petroleum derived succinic acid in a few applications such as solvents and lubricants, de-icer solutions, cosmetics, food and pharma. Applications such as 1,4-Butanediol (BDO), PBS, plasticizers, polyesters polyols market will accelerate the future growth of the market.

PBS as a key product line is used in a range of applications, including single-use in food service ware (such as cutlery and cups and lids), agricultural mulching films, and compostable bags. PBS can also be made into composites by filling the fibres with other materials to augment the material properties of the substance and, thereby, diversify the potential applications. It also finds use in pesticides and fertilizer. In the medical industry, PBS is used as biodegradable drug encapsulation systems and is also being investigated for application in medical implants. Hence, the increasing potential applications of PBS in different industries are expected to drive the demand for succinic acid for the manufacturing of PBS⁸.

The Global Bio-succinic Acid market is valued at \$1 billion in 2020. Sugarcane is the dominant Raw Material among Bio succinic Acid Market. 1,4-Butanediol (BDO) is the dominant application. Europe is the largest user of Bio succinic Acid⁹.)

It is expected that the succinic acid technology developed by the PERCAL consortium will be co-developed with an existing producer or user and the technology will be licenced. It is the use of MSW and subsequent feedstock conversion that offers potential competitive pricing and unique properties of the molecule.

⁸ https://www.marketsandmarkets.com/Market-Reports/succinic-acid-market-402.html

⁹ https://www.researchandmarkets.com/reports/5028442/global-bio-succinic-acid-market-size-forecast-to#rela4-4778591.

3.4 Biosurfactants

3.4.1 Safety & Regulatory framework

The biosurfactant has been produced by chemical and/or microbiological modification of protein and lipid fraction from the remaining fraction after fermentation of the MSW hydrolysates (to produce bioethanol, SA and LA). The obtained biosurfactants can be directly used in standard formulations of detergents as final products and are considered as a polymers according to REACH. Thus, as it was described previously, the polymers are exempted of registration obligations and the monomers > 2% included in them are the substances that must be registered. At this regard, the aminolipids obtained in PERCAL are being used in the formulation of standard detergents to substitute conventional surfactants

The EU directives and regulations that apply to BIOSURFACTANTS are indicated in the following points:

- Regulation (EC) No 648/2004 on detergents. This regulation put relevant emphasis on the biodegradability of surfactants in detergents and also the restrictions or bans on surfactants on grounds.
- FprEN 17035 Surface active agents Bio-based surfactants Requirements and test methods Approved in Jun2020. This document sets requirements for bio-based surfactants in terms of properties, limits, application classes and test methods.
- CEN/TR 16721:2014 Bio-based products Overview of methods to determine the bio-based content. This Technical Report gives an overview of methods which can be used for the determination of the bio-based content of solid, liquid and gaseous products.
- EN 16575:2014 This European Standard defines general terms to be used in the field of bio-based products, including horizontal aspects relevant for bio-based product standards.
- EN 16785 Bio-based content. This European Standard specifies a method of determining the bio-based content in products.
- Regulation (EC) No 1907/2006 concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH). According to the REACH compliance of PERCAL surfactant, aminolipid as a surfactant is not considered a substance of very high concern, it is safe handling and is not a hazard compound to the health neither to the environment.

3.4.2 Market overview

According to a new report published by Allied Market Research¹⁰ the global surfactants market was valued at \$43,655 million in 2017, and is projected to reach \$64,408 million by 2025, registering a CAGR of 5.4% from 2018 to 2025. Likewise, the global biosurfactant market¹¹ is expected to grow at a compound annual growth rate of 5.4% from 2019 to 2020 to reach USD

¹⁰ <u>https://www.alliedmarketresearch.com/press-release/surfactants-market.html</u>

¹¹ https://www.grandviewresearch.com/industry-analysis/biosurfactants-industry

2.3 billion by 2020. Biosurfactants market is a fragmented industry, owing to the presence of large number of small and medium-sized companies operating in the market. Some of the key manufacturers¹² operating in the global market are Boruta Zachem SA, AkzoNobel N.V., TeeGene Biotech Ltd., BASF, Biotensidon GmbH, Evonik Industries, Kao Corporation, Saraya Co., Ltd., Croda International, Ecover, Lion Corporation, Givaudan SA, Henkel Corporation and Jeneil Biotech, Inc.

Surface-active agents or surfactants, are organic chemicals with a hydrophilic and a hydrophobic end that when added to a liquid, change its properties at the surface or interface. The global surfactants market is classified into cationic surfactant, anionic surfactant, nonionic surfactant, amphoteric surfactant, and others. In the frame of the PERCAL project, non-ionic amino lipid surfactant has been produced. It is noteworthy that anionic surfactant and nonionic surfactant segments occupied up to two-thirds share of the surfactants market, in terms of volume in the year 2017.

By application, the surfactants market is divided into household detergent, personal care, industrial & institutional cleaner, food processing, oilfield chemicals, agricultural chemical, textile, plastic, paint & coating, adhesive, and others. The household detergent and personal care segments, together held more than two-thirds share in the surfactants market in 2017. The surfactants incorporated in detergents and soaps mix with water, and attach themselves with the dirt on clothes and other cleaning surfaces. This helps reduce the surface tension and removes the dirt from the concerned surface.



Figure 7. Global biosurfactant market revenue share, by application, 2013 (%)

North America and Europe jointly accounted for half of the market, in 2017, however, Asia-Pacific alone captured more than one-third share of the surfactants market. The prices of surfactants are comparatively high in Europe due to regulations of Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH¹³).

¹² https://www.gminsights.com/industry-analysis/biosurfactants-market-report

¹³ REACH Legislation - ECHA (europa.eu)

Based on product type, the non-ionic natural surfactants segment of the bio-based surfactants market is projected to register the highest CAGR during the coming years. The growth of this segment can be attributed to their end use in fabric softeners, shampoos, and body wash, which is increasing with the growth in the textile and personal care industries. Based on application, personal care is projected to be the fastest-growing application of natural surfactants (bio-based surfactants) during the forecast period. Increasing consumer awareness of the usage of bio-based ingredients is expected to drive the demand for natural surfactants (bio-based surfactants) in this segment. Indeed, the PERCAL biosurfactant has been tested in the household detergent applications (for dish, floor and clothes detergent applications).

Europe accounted for the largest share of the natural surfactants market (bio-based surfactants) in 2016, followed by North America. Increased demand for natural surfactants (bio-based surfactants) from Europe can be attributed to the rapidly growing applications, such as detergents, personal care, and oilfield chemicals. The demand for less toxic and highly biodegradable products due to rising concerns regarding environmental protection is the major reason for the high demand for natural surfactants (bio-based surfactants) in Europe.

Time-consuming and expensive legislative requirements are seen as a major restraint in the natural surfactants market (bio-based surfactants). Several manufacturers develop newer products to be competitive in the market, but compliance with the regulatory mandates for every product would be time-consuming and costly.

4. PERCAL Environmental assessment

The valorisation of urban biowaste developed in PERCAL project clearly contributes to the Bioeconomy Action Plan (BAP) (COM(2018) 673), recently supported by the new EU Circular Economy Action Plan published in March 2020 as part of the EU Green Deal. As stated by the Circular Economy Action Plan the transition to a more circular economy, where the value of products, materials and resources is maintained in the economy for as long as possible, and the generation of waste minimised, is an essential contribution to the EU's efforts to develop a sustainable, low carbon, resource efficient and competitive economy.

From an environmental point of view, the bioeconomy, the renewable segment of the circular economy, can turn bio-waste, residues and discards into valuable resources, which will contribute to climate mitigation and a carbon neutral future by reducing emissions and our dependence on fossil resources. In particular, as stated by the Bioeconomy Action Plan, cities should become major circular bioeconomy hubs. Circular urban development plans could translate into very significant economic and environmental gains. The Plan¹⁴ also encourages the creation of biorefineries: "The transformation towards sustainable, healthy, resource-efficient, resilient, circular and inclusive food and farming systems needs to accelerate. This includes turning organic waste, residues and food discards into valuable and safe bio-based products, for instance by deploying biorefineries. (...) Overall, actions will contribute to the

¹⁴ Bioeconomy Action Plan (BAP) (COM(2018) 673)

diversification, development and deployment of bio-based solutions and will seek to facilitate the development of new sustainable biorefineries". Within the new biorefineries to be created, urban biorefineries such as the one represented by PERCAL, will have not only a significant environmental impact but also important social and economic impacts, since currently more than 100 million tons of urban biowaste are generated every year in Europe. This new biorefineries will turn organic waste from a societal problem into a valuable resource for the production of bio-based products.

As we have been discussing in the previous sections, bio-based products have now a good opportunity to access the market. However, besides the relevance of the techno-economic feasibility of the production of the new non-fossil based counterparts, it is important to effectively demonstrate the impact of the individual processes and also the global one on the environment, through the assessment of the reduction of the carbon footprint and the GHGs emission.

PERCAL's techno-economic and Life Cycle Assessments of each individual process, comparisons and relations have been performed at the later part of the project, in order to ensure that all the process parameters and results were included, and were the most adjusted ones, considering the current TRL of some of the processes (mostly TRL 5). The base case scenario considered has been the landfilling of OFMSW together with the production of the fossil-counterparts of the selected products (Figure 8).



Figure 8. Base case (current scenario)

The functional unit for the comparisons has been set up as the unit required to treat 1,000 kg of dry OFMSW. In the base case scenario, this amount of OFMSW is going to landfill while at the same time the conventional counterparts of the selected end-products of the PERCAL project are produced in quantity equal to the one estimated from the calculations for the biorefinery development. The conventional counterpart for bioethanol, succinic acid, lactic acid, biosurfactants, hot melt adhesives (HMAs), ethyl-lactate and polyurethane urea dispersions (PUD) are gasoline, fossil-based succinic acid, lactic acid from glucose that is derived from corn, fossil-based surfactants, fossil adhesives, hexane as solvent for replacement and adipic-based PUD.



Figure 9. Alternative scenario (PERCAL Biorefinery Development)

The alternative scenario (Figure 9) proposed by PERCAL includes the biorefinery development using 1,000 kg of dry OFMSW for the production each one of the end-products, simultaneous production of biosurfactants and finally treatment of the remaining residues via landfilling. Table 1 presents the greenhouse gas emissions of the conventional products, while Table 2 presents the estimations for the greenhouse gas emissions for both scenarios for each intermediate and final product.

Conventional counterpart	GHG (kg CO2-eq/kg product)			
Gasoline	2.3			
Fossil-based succinic acid	1.89			
Lactic acid from corn	0.38			
Fossil adhesives	5.0			
Solvent (hexane)	1.0			
Adipic-acid PUD	3.4			
Fossil-based surfactants	2.1			

Table 1. Greenhouse gas emis	sion of the conventional products
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Table 2. GHGs and savings for conventional and biobased intermediates (Ethanol, Succinic acid, Lactic Acid) and final products (Biosurfactants,HMAs, Ethyl-lactate, Polyurethane dispersions) developed in PERCAL project

	Ethanol		Succinic acid		Lactic acid		Biosurfactants	
	Base Case	PERCAL	Base Case	PERCAL	Base Case	PERCAL	Base Case	PERCAL
GHG*	4,681	3,238	4,449	2,767	4,390	2,789	4,370	3,894
GHG Savings*	1	,443	1	,682	1	,601		476

(*) GHG and GHG Saving in kg CO2-eq/t OFMSW treated

	HMAs		Ethy	I-lactate	PUD	
	Base Case	PERCAL	Base Case	PERCAL	Base Case	PERCAL
GHG*	4,747	3,551	4,479	3,648	5,720	4,033
GHG Savings*	1,196		831		1,687	

(*) GHG and GHG Saving in kg CO2-eq/ t OFMSW treated

For all the products assessed a reduction in GHG emissions has been achieved. These results demonstrate the potential of the production of bio-based chemicals from waste in the fight against the GHG emissions. Changing the current chemical production routes into a more sustainable ones, as the ones proposed in PERCAL is possible and beneficial, both for the industry and for the environment.

5. Conclusions

The European Commission together with national government are working hard to advance in a favorable policy and regulatory framework which fosters the introduction of new bio-based products and technologies. The European Green Deal is the key initiative to support and boost the circular economy and the achievement of a more sustainable Europe in the future years.

Although fossil-based chemicals still control the market, the new policies and initiatives promoted from the EU, and also as a worldwide trend, which includes a great participation from the industry, have opened an opportunity for bio-based products to access the market and to start the process of substituting the fossil-based products. This is key to reduce our dependence on petroleum.

PERCAL project has shown the potential of the OFMSW to develop a platform for bio-based chemical building blocks which may in a near future, substitute part of the current fossil-based commercial products. As described, there is still room for further developments, in order to assure key factors endorse a sustainable and technoeconomic feasible production.

The assessment of PERCAL products from a regulatory point of view has provided an overview of the main considerations for the introduction and use of these products in the market. PERCAL intermediates and products obtained from OFMSW, despite coming from waste, should not be considered as waste for the envisaged applications, since they comply with end-of-waste criteria. However, a statement of conformity, according to article 6 of UE 2018/851 Directive would be required first. In case of approval, they would be subjected to REACH requirements in the same way as other chemicals. In addition, PERCAL intermediates and products do not contain substances of very high concern (SVHC) or restricted substances for the envisaged uses as listed in the as Annex XVII of REACH.

This positive analysis provides a green light for moving forward with the final products development and the production upscaling, which is key to know the real technoeconomic figures of the different processes and products. This will be key for the final exploitation and the go-to-market. A successful commercialization depends on the competitiveness of the biobased products in front of the fossil-based options, which will be obviously based on quality, production costs and price and market price.

This guide has been edited by IMECAL S.L. – PERSEO Biorefinery with the support of the PERCAL Consortium partners. For further information, please visit our site (<u>www.percal-project.eu</u>) or contact us by email (<u>percal@aimplas.es</u>).

