BIO-BASED SURFACTANTS

Present and future

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ABSTRACT

Our society has lived for a very long time on the use of fossil resources as oil. Today, research is tending to look for solutions to replace products originating from fossil resources, such as surfactants, which are present in different sectors such as pharmaceuticals, industry, cosmetics.

Today, more than 15 million tons of surfactants are produced and used yearly which leads to environmental problems such as lower potency of biodegradability, eutrophication, toxicity and so on.

It is therefore necessary to find alternatives to surfactants derived from fossil resources.

Bio-based surfactants represent a good answer to address such environmental issues: these novel surfactants are extracted from biomass such as cereals, vegetables, oilseeds, co-products and waste and other materials are under investigation as algae, insects. Biomass is valorized through a biorefinery concept.

However, finding new sources of biomass to produce bio-based surfactants at large industrial scale is not trivial, and should address the following questions:

- Quality, reproducibility and cost of biomass
- Production process feasibility, scalability and cost
- Formulation
- Impact on deforestation and global warming

Research is intensifying on the subject because bio-based surfactants are part of sustainable development.

This report is focus on bio-based surfactant from biomass.

***Key words**: surfactant, bio-based surfactant, detergency, biorefinery, biomass, environment, sustainability, circular economy*

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INTRODUCTION

Nowadays more than 15 million tons of surfactants are used yearly which leads to environmental problems such as lower potency of biodegradability, eutrophication, toxicity and so on. In order to reduce these issues research on bio-based surfactant has significantly increased.

A conventional surfactant is a petroleum-derived compound, while a bio-based surfactant is produced from biomass. They both reduce the surface tension of a liquid, the interfacial tension between two liquids or that between a liquid and a solid.

Conventional surfactant production is a chemical process (petrochemical industry)

while bio-based surfactants sustainable biomass is processed through biorefinery.

Biomass is the total amount of matter formed by all living organisms in an ecosystem at any given time. Today, the progressive shortage of fossil fuels such as petrol is a major challenge, and the valorization of biomass to produce novel bio-based surfactants gains a high interest.

Despite its added value for the environment, sustainable biomass gets advantages but also weaknesses as summarized in *Table I.*

	Bio-based surfactant	
	Strengths	Weaknesses
Environmental report	X	
Sustainable production	X	
No competition with food	X	
Biomass transport		X
Circular economy	X	
Biodegradability	X	
Reproducibility of biomass lots		X
Cost of biomass	X	
Cost of the process		X
Efficiency		X
Performance		X
Biomass stability		X
Societal acceptability/market	X	
Regulation		X
Formulation	?	
Toxicity and allergy	?	

Table I: Strengths and weaknesses of bio-based surfactants

Formulation, toxicity and allergy are under investigation.

In order to be able to fully replace chemical surfactants by bio-based surfactants some points will have to be solved:

- Technology: production, efficiency, control of energy and water consumption…
- Quantity and quality of the available biomass, its reproducibility, transport, production efficiency...
- Formulation and stability of the product
- Production costs including biomass "raw material" cost.

I - CONVENTIONAL SURFACTANTS AND BIO-BASED SURFACTANTS

A - Surfactants structures

Surfactants include two parts of opposite affinity: a **hydrophobic tail** and a **hydrophilic head.** As shown in *Figure 1*, there are four types of surfactants according to the charge carried by their hydrophilic part [1]

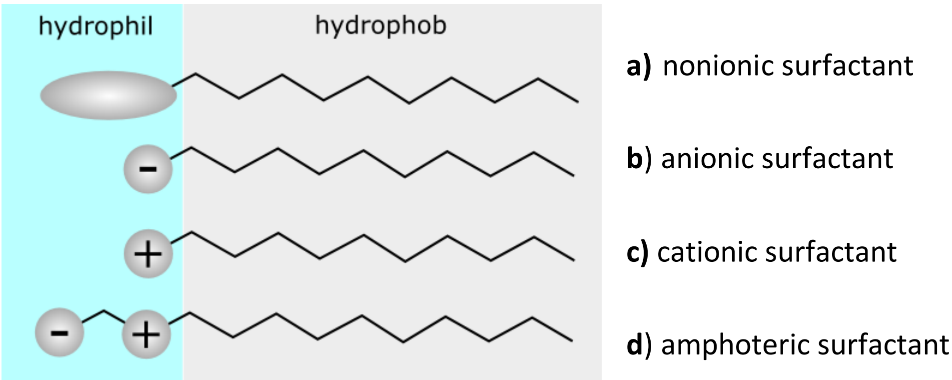

Figure 1: Structures of the different types of surfactants

- **a) Nonionic surfactants** represent around 45 % of the surfactants used in industry. They do not ionize in water or aqueous solution since their hydrophilic group is non-dissociable. In the past decade, glucoside (sugar based) head groups have been introduced to the market and exhibit a low toxicity.
- **b) Anionic surfactants** are dissociated in water in the form of a cation and an anion, generally seen in alkaline metals like (Na+, K+) or as quaternary ammonium. They are the most broadly used surfactants, representing about 50% of the world's market production.
- **c) Cationic surfactants** are dissociated in water into an amphiphilic cation and an anion. A very large proportion of this class of surfactants corresponds to nitrogen compounds such as fatty amine salts and quaternary ammoniums. They have an intrinsic antimicrobial property and are used in antiseptic, hygienic lotions…
- **d) Amphoteric surfactants** release a positive or negative charge depending on the pH of the medium. They are used as foaming agents, detergents and for body hygiene products. [2]

B - Roles of surfactants

Surfactants have different roles depending on their characteristics, as shown in *Figure 2*

- **Detergents** as indicated in part D – Mode of action of detergency.
- **Solubilizing agents** at very low concentrations, surfactants form aqueous phase solutions. When their concentration exceeds a certain value (the critical micellar concentration, CMC), the surfactant molecules are grouped into micelles. This property confers the ability for some surfactants to "dissolve" substances that are normally insoluble in the solvent used by inserting them into micelles.
- **Foaming/antifoaming agents** gas/liquid interfaces are created thanks to the presence of surfactant that absorbs to these interfaces.
- **Wetting/spreading agents** the wetting of a solid by a liquid corresponds to the spreading of the liquid on this solid. By reducing the solid-liquid surface tension, wetting agents allow the liquid to spread more easily.
- **Dispersing agents** allow to create a dispersion; usually this includes a combination of surfactants.
- **Emulsifying agents** facilitate the formation of an emulsion between two immiscible liquids. [3]

C - Specificities

Surfactant properties are characterized by their *hydrophilic lipophilic balance* (HLB).

HLB measures the degree of hydrophilic or lipophilic proportion of the surfactant, determined by calculating values for the different regions of the molecule (*Figure 2*). The HLB of a surfactant varies from 0 which is totally hydrophobic (soluble in fats and insoluble in water) to 20, totally hydrophilic (soluble in water and insoluble in fats). This value is used to predict the surfactant properties of a molecule. [4]

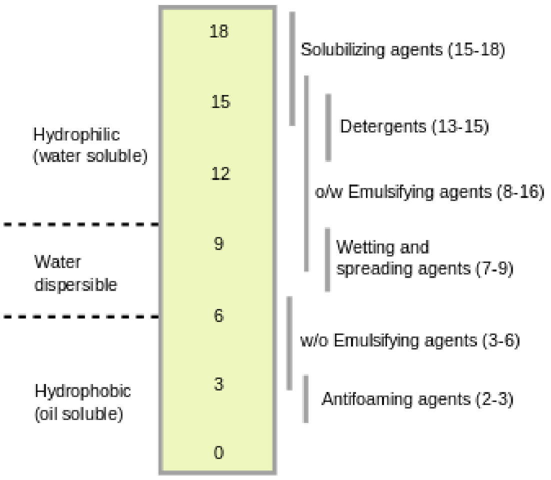

Figure 2: Classification of surfactant function according to HLB (Hydrophilic Lipophilic Balance) [5]

D - Mode of action of detergency

Detergency is the action of degreasing a surface through removal of contaminants by emulsifying them. It represents the cleansing power of a substance and is carried out in several steps.

For example, the different steps happening during a shampoo include:

- **Wetting** fat has no affinity with water and prefers to stay on the hair.
- **Dispersion** surfactant molecules disperse over the hair.
- **Agitation** the molecules are in contact with the contaminants.
- **Micellar solubilization** molecules gather around contaminants and form a micelle. The lipophilic part is fixed in the contaminant and the hydrophilic part remains in contact with the water.
- **Suspension** contaminants move away and are kept emulsified in water, trapped in the foam.
- **Elimination** contaminants are trapped in the micelles, do not redeposit on the hair and are eliminated during rinsing.

E - Key parameters to consider for bio-based surfactant production

The name bio-based surfactant is only given if at least 3 rules are respected:

- Biodegradability
- Human and Animal health
- Environment

Biodegradability

The biodegradability of an organic substance is its ability to be degraded by a biological process into smaller and less polluting molecules. Biodegradability is one of the major parameters used to assess the environmental impact of industrial substances or discharges. It prevents the long-term pollution of our soils and rivers.

Relevant tests follow standardized methods written by the OECD (Organization for Economic Cooperation and Development). Such tests make possible the evaluation of the aerobic biodegradability of a substance. Depending on the method used, different parameters such as Dissolved Organic Carbon (DOC), CO_2 production or O_2 consumption will provide valuable information on the degradation of the tested substance and are periodically measured, usually over a period of 28 days. [6]

Two biodegradabilities levels are calculated through the test:

- *The Primary biodegradability* represents the loss of surfactant properties. This must be higher than 80% in 21 days. However, this test alone does not allow to evaluate the negative impact of a detergent on the environment.
- *The Final biodegradability* represents the complete decomposition into carbon dioxide, water and mineral salts. In aerobic conditions, biodegradability must be higher than 60% in 28 days. [7]

Biodegradability tests are performed on the ingredients (surfactant / bio-based surfactant). Once the ingredient has been tested, the finished product is formulated but biodegradability is not tested again. The finish product can be a detergent, a cosmetic product...

Absence of Impacts on Human and Animal health

Since bio-based surfactants are used in many applications, they must have no negative effects on health, especially on the skin, lungs and mucous membranes. Inhalation of detergents can cause irritation of the mucous membranes of the nose and the respiratory tract. Their ingestion causes burns throughout the digestive system and can be fatal if the amount consumed is too high. [8]

They must be perfectly tolerated by the human and animal body and must have zero toxicity. Tests on health impacts must be done on the ingredient (bio-based surfactant) as well as on the finished product.

It is important to notice that the absence of toxicity of a bio-based surfactant doesn't guarantee the non-toxicity of the finished product in which it is included.

Minimal negative impact in the Environment

Conventional surfactants have harmful effects on the environment. They cause different problems as eutrophication (*Figure 3*), fish death etc. [9]

Figure 3: Effects of eutrophication on water

To comply with sustainable development, it is necessary to find molecules that have the minimal harmful impact on the environment. This will help to restore air, soil and water and limit the negative impact on fauna, flora and ecosystems.

Bio-based surfactants, derived from renewable resources, could contribute to address this issue.

F - Regulatory issues

Regulatory requirements are country-to-country dependent. Usually the REACH (Registration, Evaluation, Authorization restriction of Chemicals) rules for chemical products apply and will also concern surfactants.

II - BIO-BASED SURFACTANTS

A - Biomass

Biomass is mainly from plant coproduct and waste origin. Other biomass sources are under investigation such as algae, insects, agricultural animals, aquaculture…

Criteria for **biomass selection** include:

- Available in unlimited quantity
- Reproducible characteristics
- No competition with food resources
- Respect of environmental regulations (energy, water consumption...)

Table II. Opportunities and limitations of sustainable raw materials

[10]

Limitations	Opportunities
Heterogeneity of raw materials and contamination (pesticides, contaminants)	Definition of specifications for raw materials
Production of by-products during the extraction of raw materials	By-product valorization (biorefinery concept)
Heavy production processes, low yield, high cost	Improvement of yield, better processes
Availability/reproducibility of resources	Diversified suppliers, selection of new varieties
Agricultural practice and eutrophication problems	Sustainable agriculture: reduction of the quantity of chemicals and lower environmental impact
Questions about available areas	Valorization of fallow land
Lack of information	Information campaigns established by an organization linking farmers and industry
Global warming	Plants selection and mutagenesis

B - Origins of biomass used for bio-based surfactant production

The most well-known and used biomasses

The most well-known and best studied biomasses for the manufacture of bio-based surfactants are:

- **Hydrophilic part** is originated from carbohydrate. Compounds generally used are glycerol, glucose and sucrose from vegetable oils feedstock, sugar cane, sugar beets and starch-producing crops such as potato, maize, wheat.
- **Hydrophobic part** is originated from lipid. It is mainly obtained from vegetable oils through oilseed plants such as rape or palm oil.

Research is ongoing on novel biomass sources such as algae, insects and lignocellulosic biomass. However, problems are encountered in large-scale production, which prevents maximum exploitation of these resources.

With the diversity of biomass, a wide variety of secondary materials (co-products, waste) are generated throughout the exploitation, harvesting, collection, processing and use stages. Depending on their origin, different types of co-products and waste are under investigation [11]:

- Household and municipal waste
- Industrial waste
- Agricultural co-products

These secondary materials are used to generate economic, environmental and social gains (additional compensation for sectors, reduction of pressure on the resource and certain environmental impacts, etc.). Their valorization makes it possible to enter into the circular economy which is part of sustainable development.

Options are being explored to obtain molecules of interest from materials previously destined for disposal (e.g. chemical molecules derived from waste oils).

C – Biomass transformation process

Biorefinery is defined as a complete process transforming sustainable biomass into a range of bio-based products (chemicals, materials, food and feed...) and bio-energy (fuels, electricity, heat...).

Figure 4: Example of biorefinery

The concept of biorefinery is to strategically remove waste and recycle it by mimicking cellular metabolism, known as "industrial metabolism". [12]

As shown in *Figure 5*, the starting biomass can be cereals, oilseeds, sugar beets, wood, etc. Biorefineries processes are carried out in several steps: extraction, separation, and fractionation. This represents the first transformation of biomass. After functionalization of the biomass, intermediate agro-industrial products are obtained.

To obtain bio-based surfactants, there is still a formulation step that represents the second transformation of the biomass. The biorefinery global concept includes all the steps.

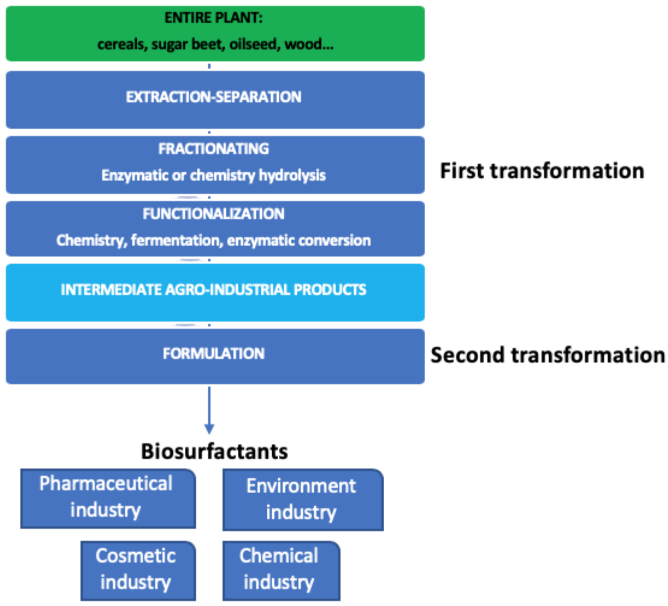

Figure 5: Processing steps in a biorefinery

Depending on the biomass used (palm oil, beets...) some steps such as extraction, purification will be different. Biorefinery can combined physico-chemical and enzymatic steps. It is therefore necessary to adapt the biorefinery to the biomass used.

D - Summary: Sources of Biomass and Surfactant Production processes

Today, bio-based surfactant production research is more focused on **plant biomass**, mainly oilseeds and cereals. As seen above, alternative biomass sources are under investigation such as algae, insects, lignocellulosic products, co-products and waste.

Constraints exist within biorefinery's mode of action. It is necessary to succeed in developing flexible tools capable of using all the plant's components and thus valorizing agricultural waste and by-products. This concept is fundamental for **circular economy**. The final objective is to develop **flexible production units** capable of using a variety of biomass to produce a multitude of products: molecules, materials...

To compete with conventional surfactants, the amount of biomass available must be large enough to allow large-scale bio-based surfactant production. To date, the quantity obtained for bio-based surfactants is often less than that required for industrial large-scale production levels. Using algae as an example, with a global production around 15,000 tonnes per year, this represents only a very small fraction of the global conventional surfactant production of 15 million tonnes per year [13]. This production is in progress due to the improvement of biomass process. Agricultural crop residues, with their significant volume, give serious hope for real perspectives of valorization of bio-based molecules.

The biorefinery process will required in the future to take into account three main pillars: economy, environment and society.

III - APPLICATIONS

There are different applications for bio-based surfactants according to the industry:

- For the *chemical industry*, the different applications are: laundry, paint, anti-corrosion coating, removal of oil and petroleum contamination, polymer synthesis.
- For the *pharmaceutical industry:* antimicrobial activity and formulation.
- For *cosmetic industry*: hygiene, care products and formulation.
- For *environment industry*: soil and water bioremediation. [14]

IV - COMMERCIALIZED BIO-BASED SURFACTANTS

Market for bio-based surfactants address two different types of customers: *producers* and *end users*.

The non-exhaustive *Annex 1 and 2* includes companies that manufacture surfactants and/or bio-based surfactants. There are four categories of end users numbered from A to D. [15]

A: Household detergents - detergents, softeners, dishwashing products

B: Market for technical and agricultural industries (paint, plastic…)

C: Industrial detergents - laboratories, hospitals …

D: Personal hygiene and cosmetics: rinsed (shampoos) and white (creams)

Annex 1 is a non-exhaustive list of companies with at least 250 users. *Annex 2* is a non-exhaustive list of companies with less than 250 employees.

Thirty-three large companies have been identified (*Annex 1)* that are producers and end users. Only seven companies produce only finished products. The others are often producers and end users.

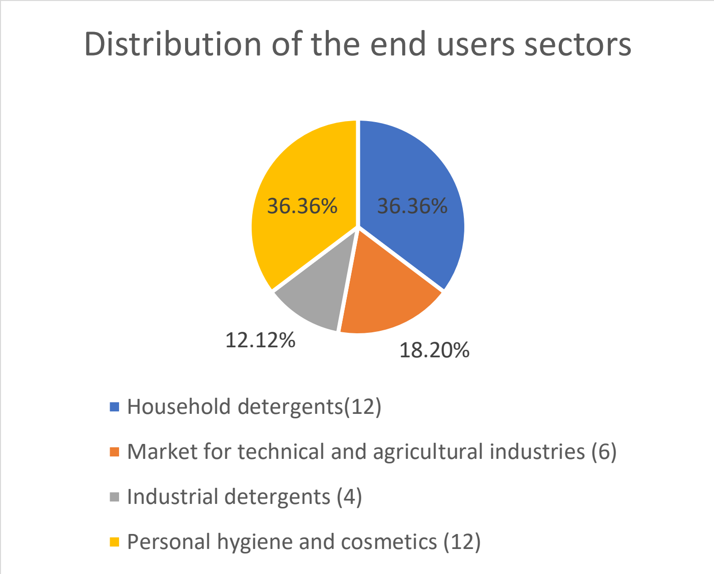

Figure 6: Distribution of the end users sectors for companies with at least 250 employees

Among the 33 companies (*Figure 6*),

- 12 manufacture products for household detergents
- 6 manufacture products for market for technical and agricultural industries
- 4 manufacture industrial detergent products
- 12 for personal hygiene and cosmetics

Concerning the seven small companies, they are almost all producers of bio-based surfactants. *(Annex 2*)

Here some example of marketed products using bio-based surfactants from sustainable origin:

- *Wheatoleo's Appyclean* which is an alkyl polyglycoside.
- *Seppic's Oramix™* which is a sugar ester. [16]
- *Bioflore's product "mousse de sucre"*, derived from corn sugar and coconut oil [17]
- *Croda's ECO* product [18]

V - DISCUSSIONS AND PERSPECTIVES

Nowadays one of the problems of our society is the progressive shortage of fossil resources such as oil used in many applications including the production of surfactants, fuel etc. It is therefore necessary to find alternative resources.

One answer to this problem is the use and valorization of sustainable biomass. It can be the future of our planet, because biomass is a natural ressource, biodegradable and non-toxic. It therefore presents arguments in favour of the transition to the use of fossil resources.

As mentioned above in *part II.B*, there are different exploitable biomasses under development:

- **Algae** have real advantages. They are currently being used in biofuel production. However, problems appear and limit large-scale production. One of the disadvantages of algae is the cost of production. Process research is underway and millions of tones are expected to be produced in the future. [19]

- **Insects** may replace microorganisms in biorefineries. The principle is based on a mass production of larvae that will be used to extract and purify ingredients of interest that can be functionalized. Barriers to the use of this biomass are known as: the consumption of water and energy to produce larvae and the uncomplete valorization of these larvae. This biomass has a great potential, but problems need to be solved to allow its industrial use. [20]

- **Lignocellulosic biomass** is another option for the future. It represents an important source of biomass and will have to be better exploited in the biorefinery concept. However, for the time being, the yield is low and its biomass is difficult to exploit on a large scale due to problems linked to the degradation of monosaccharides during glucose extraction.

- **Waste and co-products** management is a topical issue. The laws on energy transition for green growth have set environmental objectives such as: reducing fossil energy consumption by 30% by 2030, favouring the circular economy and maximising the energy potential of non-recyclable waste. This branch must continue to be exploited because it is a great interest for sustainable development. [21]

In summary, to remove the locks that block industrial production of bio-based surfactants, research must continue and will need efforts for the scaling-up and the production.

Each biomass has its own production barriers. Concerning biomass cultivated in the fields, if there is intensive cultivation of resources, this could lead to a significant risk of depletion of the organic and mineral quality of the soils. In addition, this intensive cultivation would result in the non-recommended use of fertilizers and pesticides to improve yields, which is obviously not part of a sustainable development approach.

Today, the most widely used biomass for the manufacture of bio-based surfactants is **palm oil.** Palm oil has several applications among them nutrition, pharmaceutical and so on

Figure 7: The deforestation

In order to meet an increasing demand, millions of hectares of forests have been cut down for intensive palm oil cultivation (*Figure 7).* Deforestation has a major impact on global warming, since felling and burning trees produces large amounts of greenhouse gases, particularly CO_2. This also leads to a decrease in animal biodiversity and ruin several ecosystems, for example as observed with arboreal great apes like Orang-Utans.

Research on the use of other biomass to produce lipids is facing difficulties in replacing palm oil, which is still essential today. This problem does not only concern manufacturers but also ecologists, governments, population.... This is why various organizations are being created to find solutions to stop or reduce the use of palm oil.

Sustainable palm oil cultivation

RSPO (Rountable on Sustainable Palm Oil), an organisation created in 2003, bringing together palm oil producers and distributors, scientists and NGOs (Non- Governmental Organizations) , has created in 2011 a label certifying that palm oil crops are not grown at the expense of primary forests (intact forests or forests with a high degree of naturally having never been destroyed, either directly or obviously affected by human activity). This represents a first step towards sustainability, since the protection of secondary forests is not taken into account.

Preservation of Biodiversity

An international NGO, the *Rainforest Alliance*, which works to conserve biodiversity and ensure sustainable livelihoods for local people, has created *Rainforest Alliance training and certification programs* that comply with the Sustainable Agriculture Network (SAN) and aim to achieve sustainable agriculture. The norm of sustainable agriculture is rooted in the principles of conserving natural resources and conserving biodiversity. This provides a credible solution that satisfies the most demanding criteria for palm oil cultivation. However, no real alternative exists to this day to compensate the utilization of palm oil. [22] [23] Research is in progress to find a new exotic biomass.

Other problems need to be taken into account more generally in the production and reproduction of biomass. One of the most important is **global warming**. It started about 100 years ago, and since the Industrial Revolution, average temperatures on Earth have only increased. The origin of this change is therefore linked to human activity. This change affects the entire world population and has dramatic consequences worldwide.
Industrial companies are sensitive to theses issues:

- Changes in ecosystems and resources.
- The multiplication of natural disasters.
- The transformation of climate models.
- Regulation changes.
- Improve companies attitudes to face and efficiently answer to these new environmental challenges ("green" images). [24]

All these reasons directly or indirectly affect biomass. Manufacturers will therefore have to adapt to these changes by finding other biomasses, more resistant, more adapted to these changes and in sufficient quantity to fulfil large scale industrial needs.

ACRONYMS

APG: Alkylpolyglucoside
CMC: Critical Micelle Concentration
CO_2: Carbon Dioxyde
DOC: Dissolved Organic Carbon
HLB: Hydrophilic Lipophilic Balance
NGO: Non-Governmental Organization
OECD: Organisation for Economic Cooperation and Development
O_2: Oxygen
REACH: Registration, Evaluation, Authorization, restriction of Chemicals
RSPO: Roundtable on Sustainable Palm Oil

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***Annex 1**: Examples of Producers and End users of surfactants/bio-based surfactants in companies with at least 250 employees*

	Producers	End users			
Name of companies		**A**	**B**	**C**	**D**
AkzoNobel	X		X		
ARKEMA	X				
BASF	X				
Christeyns	X	X	X	X	X
Church & Dwight		X			X
Clariant	X				
Clorox		X	X		
Colgate-Palmolive		X			X
Croda	X				
Dalli Group	X	X			X
Dow	X				
EOC group	X				
Evonik	X				
Givaudan	X				
Henkel	X	X	X	X	X
KLK Kolb	X				
L'Oréal					X
Lankem	X				
Lavo	X	X			
McBride		X			X
Naturex/Givaudan	X				
Novozymes	X				
Oleon	X				
Pilot Chemical	X				
Procter & Gamble	X	X			X
RECKITT BENCKISER	X	X	X	X	X
Roquette	X				
SEPPIC	X				X
Stanhome					X
Stepan	X				
Unilever	X	X			X
UNIVAR	X				
WERNER & MERTZ		X	X	X	

Annex 2*: Examples of Producers and End users of surfactants/bio-based surfactants in companies with less than 250 employees*

	Producers	**End users**			
Name of companies		**A**	**B**	**C**	**D**
Afyren	X				
Bioflore					X
Jeneil	X				
NatSurFact	X				
Sironix Renewables	X				
Surfact'Green	X				
Wheatoleo	X				

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