

Ecodesign of cosmetic formulae: methodology and application

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Received 22 November 2017, Accepted 29 January 2018

Keywords: biodegradability, chemical analysis, ecodesign, formulation, water footprint

Abstract

OBJECTIVE: This article describes an easy-to-use ecodesign methodology developed and applied since 2014 by the L'Oréal Group to improve the sustainable performance of its new products without any compromise on their cosmetic efficacy.

METHODS: Cosmetic products, after being used, are often discharged into the sewers and the aquatic compartment. This discharge is considered as dispersive and continuous. A consistent progress in reducing the environmental impact of cosmetic products can be achieved through focusing upon three strategic indicators: biodegradability, grey water footprint adapted for ecodesign (GWFE) and a global indicator, complementary to these two endpoints. Biodegradability represents the key process in the removal of organic ingredients from the environment. GWFE is defined herein as the theoretical volume of natural freshwater required to dilute a cosmetic formula after being used by the consumer, down to a concentration without any foreseeable toxic effects upon aquatic species. Finally, the complementary indicator highlights a possible alert on formula ingredients due to an unfavourable environmental profile based on hazard properties: for example Global Harmonization System/Classification, Labelling and Packaging (GHS/CLP) H410 classification or potential very persistent and very bioaccumulative (vPvB) classification.

RESULTS: The ecodesign of a new cosmetic product can be a challenge as the cosmetic properties and quality of this new product should at least match the benchmark reference. As shown in the case studies described herein, new methodologies have been developed to maximize the biodegradability of cosmetic formulae, to minimize their GWFE and to limit the use of ingredients that present an unfavourable environmental profile, while reaching the highest standards in terms of cosmetic efficacy.

CONCLUSION: By applying these methodologies, highly biodegradable products ($\geq 95\%$ based on ingredient composition) have been developed and marketed, with a low GWFE. This new ecodesign approach shows that embedding sustainability in developing new cosmetic products can also open up new innovative opportunities.

Résumé

OBJECTIF: Cet article décrit une méthodologie d'écoconception, facile à utiliser, développée et appliquée depuis 2014 par le groupe

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L'Oréal pour améliorer la performance durable de ses nouveaux produits, sans aucun compromis sur leur efficacité cosmétique.

MÉTHODE: Les produits cosmétiques, après utilisation, sont souvent rejetés dans les égouts et le compartiment aquatique. Ce rejet est considéré comme dispersif et continu. Un progrès important dans la réduction de l'impact environnemental des produits cosmétiques peut être atteint en se concentrant sur trois indicateurs stratégiques: la biodégradabilité, l'empreinte eau grise adaptée à l'écoconception (GWFE) et un indicateur global complémentaire à ces deux paramètres. La biodégradabilité représente le processus clé dans l'élimination d'ingrédients biologiques de l'environnement. GWFE définit ici le volume théorique d'eau douce naturelle nécessaire pour diluer une formule cosmétique, après avoir été utilisée par le consommateur, à une concentration sans effets toxiques prévisibles sur les espèces. Enfin, l'indicateur complémentaire met en évidence une possible alerte sur les ingrédients de la formule en raison d'un profil environnemental défavorable basé sur les propriétés de danger: par exemple sur le Système Général Harmonisé / Classification, Étiquetage et Emballage (SGH / CLP), classification H410 ou potentiel classement très persistant et très bioaccumulable (vPvB).

RÉSULTATS: L'écoconception d'un nouveau produit cosmétique peut être un défi car les propriétés cosmétiques et la qualité de ce nouveau produit devraient au moins correspondre au produit de référence. Comme indiqué dans les études de cas décrites ici, de nouvelles méthodologies ont été développées pour maximiser la biodégradabilité des formules cosmétiques, minimiser leur GWFE et limiter l'utilisation des ingrédients présentant un profil environnemental défavorable, tout en atteignant les plus hautes exigences en termes d'efficacité cosmétique.

CONCLUSION: En appliquant ces méthodologies, des produits hautement biodégradables ($\geq 95\%$ selon la composition des ingrédients) ont été développés et commercialisés, avec une faible GWFE. Cette nouvelle approche d'écoconception montre que l'intégration de la soutenabilité dans le développement de nouveaux produits cosmétiques peut également ouvrir à de nouvelles opportunités d'innovations.

Introduction

Water is the main environmental compartment exposed to cosmetic products. When used by the consumer, a more or less important amount of cosmetic formulae is released to the aquatic environment, in particular for rinse-off products.

Hence, in the regulatory risk assessment of ingredients, cosmetic formulae go down the drain after use by the consumer and this

release is considered as dispersive and continuous [1–3]. Whatever the cosmetic product (shampoo, hair dye, skin care, make-up, ...) is, a 100% release down the drain of the annual tonnage used by consumers, identically distributed over the 365 days of the year, must be applied by default to predict concentrations of cosmetic ingredients in the aquatic compartment [4]. Furthermore, life cycle assessment (LCA) and water footprinting following the principles of the Water Footprint Network [5] have also been applied to cosmetic rinse-off products. These studies have shown that, regarding water issue for rinse-off products such as shampoos, shower gels or soaps, one of the main contributing life cycle stages is the end-of-life of the formula, that is the discharge of remaining ingredients into the environment after the use of the cosmetic product by the consumer [6–8].

Ecodesign is an approach to create or renovate a product where special attention is paid to the environmental impacts during its entire life cycle. Ecodesign principles are defined in the International Organization for Standardization (ISO) 14062 standard [9]. This process is geared towards generating a competitive advantage by finding the best solution related to consumer needs (functionalities, cost), technical feasibility and environmental footprint. Different trials have previously been described for assessing the environmental impact of chemical-based products when discharged into the environment, through different methods or models, such as the chemical footprint [10,11], USEtox® [12] or Environment Safety Check (ESC) [13].

In 2013, L'Oréal Group launched a programme named '*Sharing Beauty With All*' [14] including a pillar dedicated to sustainable innovation of cosmetic products. The Group committed to improve the sustainable performance of all its new products put on the market by 2020 by reducing their environmental footprint or providing a social benefit. Four criteria have been retained to achieve this commitment: reduction in the aquatic environmental impact of the formula; use of renewable ingredients sustainably sourced or deriving from green chemistry; improvement of the environmental profile of the packaging; and positive social impact of the commercial product. At least one of these four criteria must be met to fulfil the above-mentioned commitment.

This article describes an easy-to-use methodology developed to address the first criterion: how to reduce the aquatic environmental impact of new formulae, in order to obtain by 2020 the highest possible percentage of improved new formulae. Three environmental indicators have been selected, to cover all possible impacts of ingredients upon the aquatic environment: (i) biodegradability, representing the key process in the removal of organic ingredients from the environment; (ii) grey water footprint, reflecting herein the theoretical volume of natural freshwater required to dilute a cosmetic formula after being used by the consumer, down to a concentration without any foreseeable toxic effects upon aquatic species; and (iii) a complementary indicator, highlighting a possible alert on formula ingredients with an unfavourable environmental profile.

The eco design of a new cosmetic product can be a challenge as the cosmetic properties and quality of this new product should match the benchmark product. The methodology herein described aims at maximizing the use of biodegradable ingredients and minimizing the environmental impact of cosmetic products while reaching the highest standards in term of cosmetic efficacy. Two case studies illustrate the feasibility and relevance of this methodology. All abbreviations used in this article are listed in Table 1.

Materials and methods

Ingredients and formulae

In the cosmetic industry, a formula is usually designed as the sum of all the ingredients necessary to obtain a lotion, cream, paste, powder, etc., offering a specific benefit to the consumer.

Biodegradability indicator

Definition

Biodegradability represents the key process in the removal of organic ingredients from the environment and more particularly from water, sediment and soil. The percentage of biodegradability of a formula is established from the percentage of readily biodegradable ingredients. Ready biodegradability has been chosen for several reasons: (i) measurement of ready biodegradation potential of organic ingredients is carried out in the main environmental compartment exposed to the release of cosmetic formulae (i.e. water) with an inoculum (i.e. microorganisms) generally coming from an aquatic ecosystem; (ii) it corresponds to the ultimate degradation process of ingredients by microorganisms under aerobic conditions in a wastewater treatment plant (WWTP); and (iii) ready biodegradability is also considered as a proof of rapid and ultimate degradation in most environmental compartments including water, sediment and soil [15]. Promoting the use of readily biodegradable ingredients therefore contributes to significantly limit the potential impact of cosmetic formulae released into the natural environment over time [16].

An organic ingredient is considered as 'biodegradable' in the calculation of the percentage of biodegradability of a formula when it fulfils at least one of the following conditions:

'Ready biodegradability' determined in a test following an Organization for Economic Co-operation and Development (OECD) (OECD 301 [17] series or OECD 310 [18]) or equivalent guideline. Further information on the interpretation of ready biodegradation tests is available in the European Chemical Agency (ECHA) guidance document R7.b [15]. All ingredients measured as 'readily biodegradable with or without the 10/14-day window' are taken into account positively in the calculation.

'Ready biodegradability' determined by read-across from 'readily biodegradable' ingredients to a structurally close analogue.

Other biodegradability status, such as 'calculated as readily biodegradable' or 'calculated/measured as inherently biodegradable' is not considered as 'biodegradable' in the calculation of the percentage of biodegradability of formulae.

The biodegradability status can be directly applied to monoconstituent ingredients or ingredients composed of homogeneous constituents (named herein as homogeneous multiconstituent ingredients) such as surfactants. These two types of ingredient, when stated as 'readily biodegradable', are counted 100% 'biodegradable' in the calculation of the percentage of biodegradability of formulae.

However, the biodegradability status cannot be applied directly to ingredients composed of heterogeneous constituents (named herein as heterogeneous multiconstituent ingredients) such as plant extracts, since biodegradation tests are not adapted to conclude on the ready or inherent biodegradability of heterogeneous mixtures. Consequently, heterogeneous multiconstituent ingredients are considered 'not biodegradable' by default in the calculation of the percentage of biodegradability of formulae.

An adaptation is typically applied to take into account readily biodegradable constituents in fragrance ingredients and can be applied to some other heterogeneous multiconstituent ingredients on a case-by-case basis: the percentage of biodegradation reached at the end of a ready biodegradation test undertaken on the ingredient itself, or the fraction (percentage w/w) of constituents known to be readily biodegradable, is included in the calculation of the formula biodegradability.

Calculation of formula biodegradability

The percentage of biodegradability of formulae is calculated according to Equation (1):

$$\text{Biodegradability \%} = \frac{\sum W_{\text{measured biodegradable ing}} \times \text{Biodeg \% ing}}{\sum W_{\text{organic ing}}} \times 100 \quad (1)$$

with

Biodegradability %: % of biodegradability of the organic part of the formula (weight/weight).

W measured biodegradable ing: weight of organic ingredients that (i) are measured as readily biodegradable with or without the 10/14-day window (monoconstituent or homogeneous multiconstituent ingredients) or (ii) contain readily biodegradable constituents (fragrances or some other heterogeneous multiconstituent ingredients on a case-by-case basis).

Biodeg % ing: 100% for ingredients (i) or specific % for ingredients (ii).

W organic ing: total weight of organic ingredients.

Biodegradability is calculated from individual ingredient data only. Global measured biodegradability of a formula is not acceptable [19].

To simplify the calculation, counter-ions and hydrates are not subtracted from Equation (1) above. In addition, organic gases are excluded from the calculation. Indeed, propellant gases used in aerosol products contribute to another type of pollution, relative to air quality. Impurities present in the formula (out of cosmetic composition) are also excluded from the biodegradability calculation.

Grey water footprint – ecodesign

Definition

The Water Footprint Network (WFN) has proposed a definition of the water footprint in the Water Footprint Manual from the concept of virtual water [5]. It is split into three types of water: (i) green water: natural water (e.g. rain) consumption by plants, (ii) blue water: surface or underground water consumption linked to human activities and (iii) grey water: volume of natural water needed to absorb pollution released into aquatic environments to meet the quality criteria set by the competent authorities.

The grey water footprint is calculated with the following Equation (2) [5]:

$$WF = \frac{L}{C_{\max} - C_{\text{nat}}} [\text{volume/time}] \quad (2)$$

where

L: pollutant load, in mass/time

C_{max}: the maximum acceptable concentration, in mass/volume

C_{nat}: natural concentration in the receiving water body, in mass/volume

In an ecodesign perspective, the grey water footprint indicator has been considered as an easy-to-use representative indicator to assess the impact of formulae on water quality for improving the environmental impact of cosmetic products. The selected criterion is the ecotoxicity of ingredients, as a key parameter common to LCA (e.g. USEtox) and environmental risk assessment.

The grey water footprint (GWF) indicator applied to our ecodesign (E) methodology of cosmetic products is named herein GWFE. It is derived from the calculation of the critical dilution volume (CDV) used in the European Ecolabel for soaps, shampoos and conditioners [20,21]. GWFE can be defined as the theoretical volume of natural freshwater required to dilute a cosmetic formula, after being used by the consumer, down to a concentration without any foreseeable toxic effects on aquatic species. It takes into account two essential parameters reflecting the environmental quality of ingredients/substances discharged into the aquatic environment: biodegradability and aquatic ecotoxicity. As CDV calculation, GWFE calculation is based on the principle of additivity of the effects of each ingredient in proportion to its concentration in the formula ($\Sigma \text{GWFE}_{\text{ing}}$ in formula).

Adaptations to the grey water footprint of the WFN

Here, the selected quality criterion is the ecotoxicity of human-made, not natural substances. Furthermore, the environmental concentration depends on the location of the release and hence on the size of the receiving water compartment. Therefore, since the methodology is relevant for all cosmetic products, irrespective of the location of use, the environmental concentration is considered as 0.

Additivity principle

The GWFE calculation is based on the principle of additivity of the effects of each ingredient in proportion to its concentration in the formula. This is an adaptation of the calculation of WFN grey water footprint, for which 'When a waste flow concerns more than one form of pollution, as is generally the case, the grey water footprint is determined by the pollutant [...] that is associated with the largest pollutant specific grey water footprint' [5]. This means that 'the GWF for each contaminant (chemical substance) of concern has to be calculated separately. The overall GWF is equal to the largest GWF found when comparing the contaminant-specific GWFs' [22].

An alternative to the calculation could have been to select only the highest amount of water calculated for each chemical of the assessed formula. However, the additive calculation has been preferred, for the following reasons: (i) the selection of the highest volume would not have allowed the formula to be ecodesigned, as only the most impactful ingredient in the formula would have impacted the GWFE result of the formula; (ii) the additive calculation is the 'reasonable worst case scenario'. It takes into account a potential similarity of toxic mode of action of ingredients, leading to an addition of their effects. This additivity principle is generally applied to the regulatory hazard classification of chemical mixtures [19] and into the assessment of these mixtures by LCA (e.g. with USEtox[®]).

Ingredient calculation

The GWFE of ingredients is calculated according to Equation (3) [23]:

$$\text{GWFE ing} = \frac{DF}{TF} \times 1000 \quad (3)$$

with

GWFE ing: ingredient grey water footprint – ecodesign (L.g^{-1}).

DF: (bio)degradation factor, as defined in the CDV (without unit).

TF: (eco)toxicity factor, as defined in the CDV (mg.L^{-1}).

This calculation is carried out differently for monoconstituent and homogeneous multiconstituent ingredients than for heterogeneous multiconstituent ingredients (Table 1).

Monoconstituent and homogeneous multiconstituent ingredients

DF depends upon the biodegradability status, obtained for each ingredient after a biodegradation test, for example a ready biodegradation test according to the OECD 301 [17] series or OECD 310 [18], or an inherent biodegradation test according to the OECD 302 series [24–26] (Table 2).

TF depends upon (i) the lowest ecotoxicity data available on three trophic levels: algae, crustaceans and fish, and (ii) a safety factor (SF) applied depending on the number of chronic or acute ecotoxicity data available (Table 3).

In the absence of specific biodegradation and/or aquatic ecotoxicity data on a given ingredient, the GHS/CLP classification can be

used to estimate its DF and TF (see Table 4). DF corresponds to the worst case linked to the classification for biodegradability. TF corresponds to an intermediate and usual case linked to the classification for acute aquatic ecotoxicity. Toxicity is the geometric mean of the acute C50 range leading to an intermediate H401/H411 classification (e.g. 3.16 mg.L^{-1} for the range $1\text{--}10 \text{ mg.L}^{-1}$) according to GHS criteria. This rule is applied to the other acute C50 ranges leading to environmental classifications ($0.1\text{--}1$ and $10\text{--}100 \text{ mg.L}^{-1}$). The acute C50 of 0.1 mg.L^{-1} is the threshold below which a substance meets the T screening criterion in the PBT classification under REACH [27]. The M factor would be taken into account.

Table 2 Degradation factor (DF)

Biodegradability status	Degradation factor (DF)
Readily biodegradable measured	0.05
Readily biodegradable measured without 10-day window	0.15
Inherently biodegradable measured	0.5
Readily biodegradable calculated	0.5
Not inherently biodegradable measured	1
Not biodegradable	1
Not readily biodegradable calculated	1
Not readily biodegradable measured	1
Vegetal extract	0.5
Fragrance	0.5
UVCB except vegetal extract	1
Inorganic	1
No data	1

DF values derived from [23]. (UVCB: substances of unknown or variable composition, complex reaction products or biological materials).

Table 3 Toxicity factor (TF)

Data	Safety factor (SF)	Toxicity factor (TF)
One short-term L(E)C50	10 000	Toxicity/10 000
Two short-term L(E)C 50 from species representing two trophic levels (fish and/or crustaceans and/or algae)	5000	Toxicity/5000
At least 1 short-term L(E)C'50 from each of three trophic levels of the base-set	1000	Toxicity/1000
One long-term NOEC (fish or crustaceans)	100	Toxicity/100
Two long-term NOEC from species representing two trophic levels (fish and or crustaceans and/or algae)	50	Toxicity/50
Long-term NOEC from at least three species (normally fish, crustaceans and algae) representing three trophic levels	10	Toxicity/10

Reference: [23].

Table 1 List of abbreviations

BOD5	Biochemical oxygen demand in 5 days
CAS RN	Chemical Abstract Service Registry Number
CDV	Critical dilution volume
CoHCs	Chemicals of high concern
DF	(bio)Degradation factor
DID list	Detergent Ingredient Database
ECHA	European Chemical Agency
EC No	European Community Number
EINECS	European Inventory of Existing Commercial Chemical Substances
ELINCS	European List of Notified Chemical Substances
ESC	Environmental safety check
FIL	Full ingredient labelling
GHS CLP	Globally Harmonized System Classification, Labelling and Packaging
IB	Inherently biodegradable
INCI	International Nomenclature Cosmetics Ingredients
IUPAC	International Union of Pure and Applied Chemistry
ISO	International Organization for Standardization
GWFE	Grey water footprint
GWFE	Grey water footprint adapted for ecodesign
HsCi	Hazard statement for constituent
LCA	Life cycle analysis
MSDS	Material Safety Data Sheet
NICNAS	National Industrial Chemicals Notification and Assessment Scheme
NOEC	No observed effect concentration
OECD	Organization for Economic Co-operation and Development
PBT	Persistent bioaccumulative and toxic
PEF	Product environment footprint
PNEC	Predicted no effect concentration
ProjEC	Projected environment concentration
REACH	Registration, Evaluation, Authorisation and Restriction of Chemicals
RB	Readily biodegradable
SF	Security factor
TF	(eco)Toxicity factor
TFi	Toxicity factor for the hazard statement 'i'
ThOD	Theoretical oxygen demand
US EPA	United States Environmental Protection Agency
UV	Ultra violet
vPvB	Very persistent and very bioaccumulative
WF	Water footprint
WFN	Water Footprint Network
WWTP	Wastewater treatment plant

Table 4 DF and TF vs. GHS/CLP classification for an ingredient

Classification	Degradation factor	Safety factor	Toxicity (mg.L ⁻¹)	Toxicity factor (mg.L ⁻¹)
H400	0.05	1000	0.316	0.000316
H410	1	1000	0.316	0.000316
H401	0.05	1000	3.16	0.00316
H411	1	1000	3.16	0.00316
H402	0.05	1000	31.6	0.0316
H412	1	1000	31.6	0.0316 ¹
H413	1	1000	100	0.1

Heterogeneous multiconstituent ingredients

Many ingredients used in cosmetic products are heterogeneous multiconstituent ingredients, particularly vegetal-derived ones. In general, vegetal-derived ingredients are complex substances obtained by processing a plant or its parts by a treatment such as extraction, distillation, pressing, fractionation, purification, concentration or fermentation. The composition of these ingredients varies depending on the genus, species, growing conditions and harvest period of the sources and the technical process applied [28, 29].

When the composition of a heterogeneous multiconstituent ingredient is well known (*e.g.* essential oils), GWFE can be determined from specific data on constituents. DF can be estimated from available ready and/or inherent biodegradation test results on constituents according to Equation (4):

$$DF_{CM} = [RB\% \times 0.05] + [RB_{FW}\% \times 0.15] + [IB\% \times 0.50] + [(1 - RB\% - RB_{FW}\% - IB\%)] \quad (4)$$

with

DF_{CM}: degradation factor of the complex mixture

% RB: % w/w of readily biodegradable components in the complex mixture

% RB_{FW}: % w/w of readily biodegradable components failing the 10/14-day window criterion in the complex mixture

% IB: % w/w of inherently biodegradable components in the complex mixture

TF can be estimated from ecotoxicity data on constituents, or from their GHS/CLP classification as described below for fragrances.

When the ingredient composition is partially unknown or insufficiently precise, or when biodegradation and ecotoxicity data on constituents or their GHS/CLP classifications are inconsistent, the general GHS/CLP classification of the ingredient can be used. In this case, GWFE value is obtained from DF and TF values given in Table 4. This latter method generally penalizes ingredients because TF is often lower as compared to the one calculated from specific data on constituents, leading to a higher GWFE.

Botanically derived ingredients are considered inherently biodegradable by default and classified as H411 (reasonable worst case). DF and TF values are therefore 0.5 and 0.00316 mg.L⁻¹, respectively (see Table 4).

When a heterogeneous multiconstituent ingredient is displayed as 100% readily biodegradable (supplier's statement) and without GHS/CLP classification, a DF of 0.15 (readily biodegradable failing the 10/14-day window criterion) and a TF of 0.1 are applied.

Fragrances

Fragrances are particular heterogeneous multiconstituent ingredients since suppliers typically keep the composition confidential to preserve manufacturing secrets. The following GWFE calculation is adapted to this specificity of fragrances.

Default values for fragrance ingredients in the DID list from the European Ecolabel for soaps, shampoos and conditioners are DF = 0.5 and TF = 0.002 mg.L⁻¹ [28–30]. These values do not reflect ecoconception efforts undertaken by fragrance manufacturers. Manufacturers are therefore asked to provide for each fragrance a sustainable certificate giving the distinct percentages (w/w) of readily and inherently biodegradable constituents (assuming fragrances are systematically 100% organic), and the percentages of constituents classified or not classified according to the CLP regulation [19].

DF of the fragrance ingredient is calculated from the percentages of readily (regardless of the fulfilment of the 10/14-day window criterion) and inherently biodegradable constituents provided by the supplier and the TF from the GHS/CLP classifications of the constituents.

DF and TF values are calculated according to Equations (5) and (6), respectively:

$$DF = [RB\% \times 0.15] + [IB\% \times 0.50] + [(1 - RB\% - IB\%)] \quad (5)$$

with

RB %: w/w % readily biodegradable ingredients in the fragrance

IB %: w/w % inherently biodegradable ingredients in the fragrance.

The percentage of readily biodegradable ingredients is multiplied by 0.15 instead of 0.05 since it combines both readily biodegradable statements by measurement (see Table 2 above). As details concerning the fulfilment of the 10/14-day window criterion are generally unknown, a worse case is applied (*i.e.* failing this criterion).

$$TF = \sum HsCi \% \times TFi \quad (6)$$

with

HsCi %: w/w % of constituents with the hazard statement (Hs) 'i' in the fragrance

TFi: reference TF for the hazard statement 'i'.

Strong acids and bases

The strong acids and bases listed below are accounted in the total weight of the formula for GWFE formula calculation (see Equation 7) but with a set at 0.

In fact, these ingredients are assumed to have a negligible impact on the GWFE value because they react with other ingredients in the formula and the reaction products are considered non-ecotoxic. Table 5 lists the strong acids and bases concerned.

Propellant gases

Propellant gases are released in the air after use and therefore not taken into account in the ecodesign methodology described in this article based on water impact. Organic molecules can be photodegraded but biodegradability is much more difficult to assess in air [31]. In addition, the fraction of cosmetic propellant gases that can reach soils and/or waters (where a biodegradation potential is of interest) is generally low due to their high Henry's law constants [32]. The GWFE assessment is therefore not applied to propellant gases.

Table 5 Strong acids and bases excluded from calculation of formula GWFE

INCI name	CAS RN	EC number
Hydrochloric acid	7647-01-0	231-595-7
Phosphoric acid	7664-38-2	231-633-2
Sulphuric acid	7664-93-9	231-639-5
Potassium hydroxide	1310-58-3	215-181-3
Sodium hydroxide	1310-73-2	215-185-5
Calcium hydroxide	1305-62-0	215-137-3
Ammonium hydroxide	1336-21-6	215-647-6

CAS RN: Chemical Abstracts Service Registry Number; EC: European Community; INCI: International Nomenclature of Cosmetic Ingredients.

Volatile ingredients

Volatile ingredients, such as ethanol or some nail varnish solvents, are not excluded from the calculations of formula biodegradability and GWFE. The volatility of a solvent in a cosmetic product is different from that of a pure solvent: several parameters would have to be taken into account to determine the actual volatility such as water solubility, molecular weight, vapour pressure and/or Henry's law constant. So, predicting the volatility of solvents present in cosmetic products remains very complex. Moreover, some of the volatile solvents of nail varnishes are trapped by nitrocellulose and other polymers and serve as plasticizers (which enable the properties of the polymers in the film to being adjusted). This trapping phenomenon also exists in hairsprays.

Generic data

When no relevant data are available for an ingredient, the precautionary principle is applied using biodegradability and ecotoxicity values corresponding to a reasonable worst case: the ingredient is considered not readily biodegradable and classified as H411. This GHS/CLP classification leads to generic DF and TF of 1 and 0.00316 mg.L⁻¹, respectively (see Table 4). For vegetal extracts and fragrances, a generic DF of 0.5 is applied (see Table 2), whereas the generic TF remains 0.00316 mg.L⁻¹.

Formula GWFE

The GWFE of the formula is calculated by summing the GWFE of each ingredient and dividing by the total amount of material present in the formula according to Equation (7):

$$\text{GWFE formula} = \frac{\sum \text{Wing} \times \text{GWFE ing}}{W} \quad (7)$$

with

GWFE formula: grey water footprint – ecodesign of the formula (L.g⁻¹).

Wing: weight of the ingredient in the formula (g).

GWFE ing: grey water footprint – ecodesign of the ingredient (L.g⁻¹).

W: total weight of the formula, including water and excluding propellant gases (g).

Impurities present in the formula (out of cosmetic composition) are also excluded from the GWFE calculation.

Complementary indicator

This ecodesign methodology aims at replacing or limiting the use of ingredients with an unfavourable environmental profile. However, certain types of ingredient cannot be detected as being unfavourable through biodegradability potential and GWFE alone. Thus, the bioaccumulation potential of lipophilic ingredients should also be captured by a complementary indicator. In this way, ingredients provisionally suspected of being very persistent and very bioaccumulable (because non-readily biodegradable and potentially bioaccumulable) could be identified. In this perspective, a third complementary indicator is used. This indicator gives four possible global statements on the environmental profile of an ingredient, displayed with different colours to inform formula developers in an easily understandable way. The colour depends on the alert level on the ingredient:

- Green: the ingredient presents no alert for the environment.
- Yellow: low-level alert if an ingredient is 'suspected' of being PBT-vPvB, is H400, H410 or H411, presents a GWFE_{ing} ≥ 10⁶, or another unfavourable characteristic.
- Orange: high-level alert if an ingredient is 'highly suspected' of being PBT-vPvB, or presents another very unfavourable characteristics.
- White: no statement since any specific ingredient number or International Nomenclature Cosmetics Ingredients (INCI) name has been attributed to this ingredient for confidential reason (e.g. fragrance ingredients).

The formula developer therefore has three available indicators for ecodesigning new products with regard to their environmental impact: biodegradability % w/w, GWFE expressed as litres of water per gram of formula and a qualitative global environmental indicator.

Data sources

The three indicators used in the formula ecodesign methodology are derived from environmental data on ingredients. All ingredients appearing on the full ingredient labelling (FIL) of the marketed product packaging are taken into account to calculate these indicators.

Prioritization is given to measured data obtained from international regulatory databases: Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH) registration dossiers, National Industrial Chemicals Notification and Assessment Scheme (NICNAS) and United States Environmental Protection Agency (US EPA); data published in the 2014 Detergents Ingredient Database (DID) list [28–30]; and data from documents provided by ingredient suppliers such as the Material Safety Data Sheet (MSDS) or other external sources.

The methodology is based on water solubility, biodegradability, bioaccumulation, ecotoxicity and substance classification data. The general decision tree used to determine DF and TF values for each ingredient. It is based on available environmental data (Figure 1).

Formula ecodesign

Formula categorization

The comparison between the environmental profiles of formulae is only relevant for an identical benefit provided to the consumer. For example, a 'simple' shampoo that only provides a 'hair washing'

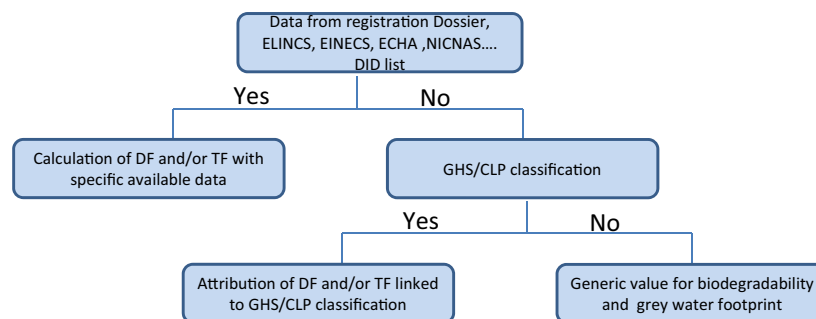


Figure 1 General decision tree for the determination of an ingredient's DF and TF.

service cannot be compared to an anti-dandruff shampoo, which provides two benefits: 'hair washing' and 'anti-dandruff action'. Therefore, a categorization based on the benefit provided to consumers has been defined. This categorization has been applied to all cosmetic products. Twenty major families including more than 140 categories based on service (also named herein as functional categories) have been defined (Table 6). The principle of categorization of commercial products by benefit provided to consumers has been supported by Cosmetics Europe [33] and is being discussed at the European Commission level.

Formula assessment

Reference values have been determined to assess the improvement of the environmental impact of new formulae. As 2013 was the launch year of the sustainable development programme 'Sharing Beauty With All' [14], it has therefore been chosen as the reference year for the further improvement of the environmental profile of new formulae. Since 2014, the environmental impact of all new commercialized formulae is compared to reference values of 2013, also named baselines in this article.

For that purpose, annual biodegradability (%) and GWFE (L g⁻¹) averages per functional category have been calculated for formulae

sold in 2013, weighted by their 2013 sale volumes, to reflect the average profile of L'Oréal Group's products on the market (Equation 8).

$$\text{GWFE functional category baseline (2013)} = \frac{\sum \text{GWFE formula of category} \times \text{sale volume}}{\text{Total sale volume of formulae in category}} \quad (8)$$

To simplify the exploitation of GWFE data by formula developers and to be able to compare the environmental profile of formulae relative to their baselines also among or even across product families, it has been decided to normalize the GWFE in base 100. In other words, the GWFE functional category baseline corresponds to 100 for all functional categories and the normalized GWFE of a given formula is obtained according to Equation (9):

$$\text{Normalized GWFE formula} = \frac{\text{GWFE formula of category}}{\text{GWFE functional category baseline (2013)}} \times 100 \quad (9)$$

The objective of formula developers is to achieve a better biodegradability and a reduced GWFE of new formulae relative to the baselines of the functional category. This target can be displayed on a graph showing biodegradability vs. GWFE (Figure 2a).

Table 6 List of functional categories defined by benefit provided to consumers – examples of shampoos and cleansers categories

Shampoos subcategories

Anti-dandruff shampoos for normal hair, natural hair or greasy hair
 Anti-dandruff shampoos for sensitized hair to very sensitized or damaged hair
 Shampoo (excluding anti-dandruff shampoo) for normal hair, natural hair or greasy hair
 Shampoo (excluding anti-dandruff shampoo) for sensitized hair to very sensitized or damaged hair
 Shampoo (excluding anti-dandruff shampoo) for children or babies

Cleansers subcategories

Make-up removers – milks/creams
 Make-up removers – aqueous textures
 Make-up removers – oily textures
 Facial washes – with exfoliating agent
 Facial washes – without exfoliating agent

Case studies

The two examples below illustrate how this methodology has been successfully applied to thousands of new formulae to make ecoconception, and more globally sustainable development, also considered as a pillar of innovation.

Shampoo (excluding anti-dandruff shampoo) for normal, natural or greasy hair

This shampoo shows a biodegradability of 96.7%, which is better than the L'Oréal's baseline of its functional category at 92.7% (Figure 2b). Therefore, improvement reaches 5%. The normalized GWFE (base 100) of this shampoo is better than the L'Oréal's baseline as well, being 11 as compared to 100. So an improvement of about 89% is obtained in this case.

The contribution of each ingredient to the biodegradability and GWFE (Table 7) has been taken into account by the formula developer to ecodesign this shampoo.

In this example of shampoo, a short part of surfactants A (0.8%) is not considered as biodegradable. The fragrance and the 'other ingredients' (C and J, respectively) do not reach 100% biodegradability as

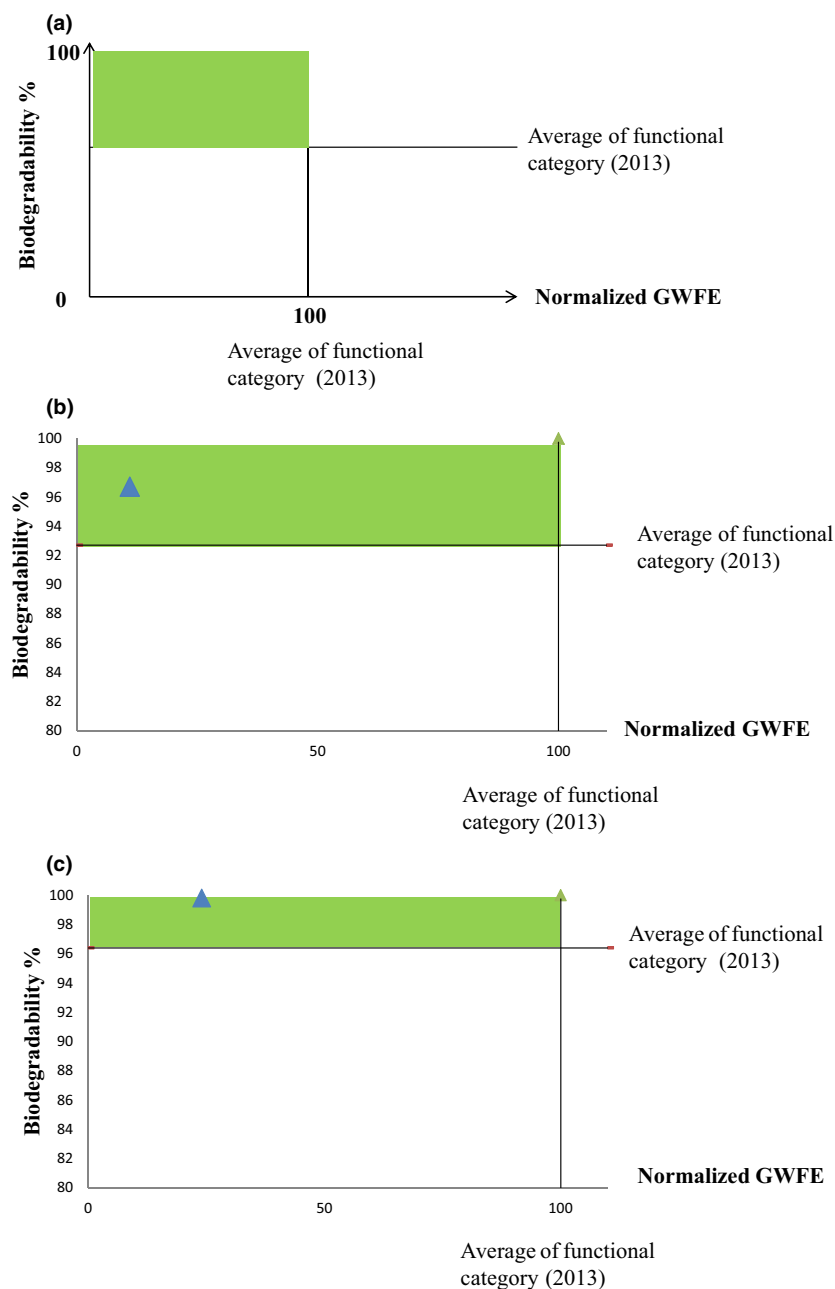


Figure 2 (a) General scheme to express ecodesign indicator values for formula compared to the 2013 average of the functional category (baselines): the green zone corresponds to an improvement of the formula. (b) Biodegradability vs. normalized GWFE for an ecodesigned shampoo belonging to the functional category shampoo (excluding anti-dandruff shampoo) for normal, natural or greasy hair. (c) Biodegradability vs. normalized GWFE for an ecodesigned cleanser belonging to the functional category facial washes – without exfoliating agent.

well. Concerning GWFE, surfactants are the most impacting ingredients. Ingredient A contributes to about 58% of the formula GWFE. Ingredient B (preservatives) is the second most impacting ingredient contributing to more than 25% of the formula GWFE.

Even if this formula has a favourable environmental profile as such (biodegradability close to 100% and low normalized GWFE, see Figure 2b), it could potentially be further improved through

limiting the concentration of some ingredients such as surfactants. But in a shampoo, surfactants are the core technology of the formula. For the same target of benefit provided to the consumer, limiting concentration of surfactants usually leads to reduction in cosmetic efficacy, a possible consequence which needs also to be put into balance to make appropriate ecodesign choices.

Table 7 Example of an ecodesigned shampoo

Ingredients	Ingredient concentration in formula	Organic %	Complementary environmental indicator	Ingredient biodegradability (%)	GWFE ing (L.g ⁻¹)	Contribution of the ingredient to the formula GWFE (%)
A (surfactants)	48.6	29.35		99.2	643	57.78
B (preservatives)	0.7	100		100	19 913	25.75
C (fragrance)	0.7	100		80.6	10 667	13.67
D (soothing agent)	0.01	100		100	500	0.01
E (pH adjuster)	qs pH	0		0	0	0
F (pH adjuster)	qs pH	100		100	31	0
G (viscosity adjuster)	0.4	0		0	1000	1.65
H (emulsifier)	0.7	100		100	100	0.13
I (water)	49.57	0		0	0	0
J (other ingredients)	0.02	61.78		59.8	48 523	0.95
FORMULA	100			96.7		100

Table 8 Example of an ecodesigned cleanser

Ingredients	Ingredient concentration in formula	Organic %	Complementary environmental indicator	Ingredient biodegradability (%)	GWFE ing (L.g ⁻¹)	Contribution of the ingredient to the formula GWFE (%)
A (surfactants)	41	37.4		100	2064	81.58
B (moisturizing agent)	5	100		100	2500	11.95
C (water)	51.11	0		0	0	0
D (pH adjuster)	0.19	100		100	30	0.01
E (pH adjuster)	qs pH	0		0	0	0
F (fluidifying agent)	0	100		100	500	0.24
G (viscosity adjuster)	2	0		0	1000	2.39
H (fragrance)	0.4	100		90.1	9000	3.55
I (preservative)	0.2	100		100	250	0.05
J (other ingredients)	0.1	100		95	9460	0.91
FORMULA	100			99.8		100

Ingredients C and J are mixtures that have a biodegradability lower than 100%. However, they do not contribute to an important decrease in biodegradability of the shampoo due to their low concentrations. The percentage of biodegradability of the fragrance could be improved from 80.6% to 100% by changing its composition, which equates to a fragrance substitution.

To improve the GWFE value, the concentration of the most impacting surfactants could be reduced – with a risk of lower cosmetic efficacy – or some surfactants could be replaced by less impacting ones if technically feasible.

Facial washes – without exfoliating agent

This cleanser shows a biodegradability of 99.8%, which is better than the L'Oréal's baseline of its functional category at 96.4% (Figure 2c). Here, improvement is 3.4%. The normalized GWFE (base 100) of this cleanser is better than the L'Oréal's baseline as well, being 24 as compared to 100. So a four-fold improvement is achieved in this case.

The contribution of each ingredient to the biodegradability and GWFE (Table 8) has been taken into account by the formula developer to ecodesign this cleanser.

In this second example of cosmetic product, the formula also has a very good environmental profile as the complementary indicator

does not indicate any specific ecodesign alert on ingredients. Reaching 100% biodegradability could only be achieved by replacing ingredients H (fragrance) and J (other ingredients) by 100% biodegradable surrogates. GWFE of the formula is mostly due to ingredient A (surfactants) with a contribution of 81%.

Discussion

Comparison with existing ecodesign methodologies

Several methodologies already exist to assess the impact of chemicals discharged into the environment on water quality. In life cycle assessment (LCA), impacts such as marine and freshwater eutrophication or freshwater ecotoxicity are widely used. But the methodologies are neither easy to implement [34] nor applicable to all cosmetic ingredients yet. For example, eutrophication only applies to cosmetic ingredients containing nitrogen or phosphorus. Furthermore, LCA considers average potential impacts on ecosystem, to highlight hotspot impacts and identify the most relevant ecodesign actions. The ecodesign methodology of cosmetic products presented herein addresses a key impact at their end-of-life cycle stage, with the objective to preserve the whole aquatic food chain in agreement with risk assessment principles.

The concept of grey water footprint, developed by the WFN, focuses on the impacts of chemicals into the aquatic ecosystems. Grey water represents the volume of natural water needed to absorb pollutants released into aquatic environments to meet the quality criteria set by the competent authorities [5]. The selected quality criteria have to be relevant to the contaminants released into water by the assessed products. Nitrogen, phosphorus, pesticides and metals [22] are criteria often used, especially for agricultural products [35]. Other criteria, such as ecotoxicity, biochemical oxygen demand in 5 days (BOD5) [9] and theoretical oxygen demand (ThOD), have also been assessed (internal data). It appears that the ecotoxicity criterion seems to be the most relevant quality criterion for the end-of-life of a cosmetic formula. Another potential ecodesign method is described in the Chemical Footprint Project. This method is less final product-oriented as compared to the ecodesign approach of cosmetic products described herein because it considers the total mass of chemicals of high concern (CoHCs) in products (including formulae and articles) sold by a company and used in its manufacturing operations or by its suppliers [36].

The ESC methodology [13], based on the regulatory risk evaluation approach, has also been proposed to assess the environmental impact of chemical-based products using the projected environment concentration (ProjEC) / predicted no-effect concentration (PNEC) ratio as an indicator. ESC is a more global approach, where aggregated formula quantities marketed per year are necessary and essential for a precise assessment in a given geographic area, contrary to the ecodesign methodology presented herein. The latter has not been created for risk assessment, but to limit and decrease the global aquatic impact of marketed cosmetic products.

Ingredient data quality and stability

Environmental data collection from thousands of existing and new cosmetic ingredients is also an extensive programme in itself but appears realistic. Many data are already available in public databases or other publicly available sources (e.g. MSDS, scientific publications). Data quality is essential and data update can be a perplexing aspect to keep track of. One notable issue is that the DID list [30] describes the ingredients with trivial names, without Chemical Abstract Service Registry Number (CAS RN), European Community Number (EC No), International Nomenclature Cosmetics Ingredients (INCI) or International Union of Pure and Applied Chemistry (IUPAC) nomenclature, which can make information analysis a challenging task. On the other hand, the abundance of data, particularly in REACH registration dossiers on ECHA website, requires expert judgement to confidently select appropriate data on ingredients (e.g. sodium lauryl sulphate). This difficulty is not a specific issue of the described methodology.

Although updated *ca.* every 5 years, the DID list data are stable. However, other data sources can change daily. When two ingredient suppliers show different results and classifications, it can be difficult to select relevant data without access to the study reports or at least detailed results. In general, the worst case is retained. For other ingredient supplier documents, there is also no guarantee that the document can be easily tracked if the Internet link changes. Concerning REACH registration data, dossier updates can take place at any time with no traceability of the changes. The only information available on the ECHA website is the date of the last update. Therefore, if an endpoint data are disputed or no longer appears in the dossier (or has changed Klimisch score), it

will be impossible to prove that the selected data are still present on the ECHA website. This point is important considering the relationship with stakeholders and for proving the sincerity and accountability of data management.

Data quality and stability issues are the same whatever the methodology used (LCA and risk assessment included). The methodology presented in this article has been set with the best possible data traceability until the data update process from databases can be automated.

Biodegradability indicator

Regarding biodegradability, the assessment approach considers aerobic biodegradation only, and it therefore differs from the CDV approach [20] where anaerobic data can also be taken into account. The choice to only take into account aerobic conditions was made to focus first on freshwater surface as a reasonable worst case scenario. Furthermore, anaerobic biodegradation data are rare with respect to the thousands of ingredients used in cosmetic products.

Determination of the biodegradation statement may be problematic for monoconstituent ingredients following chemical regulatory principles. A monoconstituent ingredient that is readily biodegradable but failing the 10/14-day window criterion is considered non-persistent in the environment according to the PBT criteria under REACH, but not rapidly biodegradable according to the GHS/CLP criteria where the 10/14-day window criterion must be fulfilled. Unfortunately, when making data analysis of monoconstituent ingredients and particularly MSDS analysis, biodegradability is often just indicated as X% in 28 days (end of the test) and the fulfilment of the 10/14-day window criterion is rarely mentioned. In the ecodesign methodology described in the present article, this potential issue is not relevant as monoconstituent ingredients are considered as 'biodegradable' for calculation of the formula biodegradability when they are readily biodegradable with or without fulfilment of the 10/14-day window criterion. Moreover, this criterion is not applied to homogeneous multiconstituent ingredients such as surfactants, as it is assumed that the lag time before degradation starts varies as a function of the carbon chain length of the different constituents in ready biodegradation tests. As mentioned in § *Heterogeneous multiconstituent ingredients* of section Ingredient calculation in Materials and methods, a heterogeneous multiconstituent ingredient cannot be considered as readily biodegradable from a test performed on the ingredient itself. All constituents must be readily biodegradable by themselves to apply this status to such an ingredient. So, the large majority of natural extracts are considered by default as 'not biodegradable' in the methodology due to poor knowledge on their composition and/or the presence of known non-readily biodegradable constituents. When useful for the assessment of the formula, the percentage of biodegradation reached at the end of a ready biodegradation test undertaken on the ingredient itself, or the percentage (w/w) of constituents known to be readily biodegradable in the ingredient, is included in the calculation of the formula biodegradability. This adaptation is typically applied to take into account readily biodegradable constituents in fragrance ingredients and can be applied to some other heterogeneous multiconstituent ingredients on a case-by-case basis. However, this process is time-consuming and/or often underestimates the biodegradation potential of heterogeneous multiconstituent ingredients. The development of a test methodology that would allow to conclude on the ready or inherent biodegradability of heterogeneous multiconstituent ingredients

directly from the test results, based for example on carbon balance evaluation, would be helpful for an appropriate and accurate assessment of complex mixtures such as plant extracts.

Formula biodegradability

A limit in the methodology relates to the improvement of biodegradability of formulae. By voluntarily adding readily biodegradable ingredients, it is possible to increase the percentage of biodegradability of the formula without any improvement of the cosmetic properties. However, formula developers are not encouraged to apply approach without any additional cosmetic benefit. Furthermore, this methodological limit is counterbalanced by an increased GWFE of the formula.

In agreement with the European Ecolabel methodology [20], inorganic ingredients (pigments, clays, metals, etc.) are excluded from the formulae biodegradability calculation. These ingredients are not biodegradable and can have an impact on the environment. The relevance of a percentage of biodegradability excluding inorganics present in the formula could therefore be questioned. However, this methodological limit is counterbalanced via the GWFE calculation that considers the lack of biodegradation and the aquatic toxicity of inorganic ingredients.

Grey water footprint – ecodesign

When compared to other methodologies, such as USEtox, GWFE can be easily implemented in a company ecodesign strategy applied to a high number of products. GWFE is also easier to apply and manage than the critical dilution volume (CDV) criterion used by the European Ecolabel. GWFE is calculated from the (acute or chronic) toxicity data on the most sensitive aquatic species, whereas the median of (acute or chronic) toxicity data must be determined on the different aquatic trophic levels prior to CDV calculation [28, 29].

The GWFE calculation is based on chronic impacts, but only few chronic aquatic toxicity data are available for cosmetic ingredients. Therefore, as for the CDV approach, the methodology allows acute aquatic toxicity data to being used for ingredients for which chronic data are lacking. A safety factor (SF) enables the extrapolation from acute to chronic data.

A toxicity factor (TF) is assigned to each ingredient of the formula based on available aquatic ecotoxicity data and a safety factor (SF) that depends on the data set (number of test species and trophic levels, acute or chronic data). The TF used in this methodology retains the lowest ecotoxicity value for the calculation. It can be more conservative than the TF applied in the CDV method that retains the median ecotoxicity value of the most sensitive trophic level for calculation. The main reason for using a methodology based on the lowest ecotoxicity value is data scarcity. It is common to find two or three acute aquatic ecotoxicity data only for example [37].

On the other hand, SF of an ingredient can range from 10 000, when one acute C50 only is available for a single trophic level, to 10, when chronic data are available for the three standard trophic levels (algae, crustaceans and fish). SF value can therefore vary by a factor of 1000. In fact, SF integrates the precautionary principle and generally penalizes ingredients with acute data only. Although relevant from the perspective of environmental protection, SF use can lead to apparently debatable ecodesign choices in some cases. These cases need to be identified when possible in order to address them better. So the same TF can be obtained from a single acute

C50 > 100 mg.L⁻¹ (maximum concentration tested) and from three chronic no observed effect concentration (NOECs), the lowest of which being 0.1 mg.L⁻¹. In the first case, the ingredient does not appear to be toxic to aquatic life, whereas in the second case, the ingredient is toxic or very toxic to aquatic life and should be classified as H410 or H411, with the GHS09 pictogram. For example, a largely used surfactant currently has the following data: H400-H411 GHS/CLP classification; lowest acute aquatic C50 = 0.5 mg.L⁻¹; lowest chronic aquatic NOEC = 0.128 mg.L⁻¹. The calculated TF from acute data is $0.5/1000 = 0.0005$ mg.L⁻¹, whereas calculated TF from chronic data is $0.128/10 = 0.0128$ mg.L⁻¹. Consequently, TF value based on chronic NOECs is more favourable and would be equivalent to TF of another surfactant with a lowest acute C50 among the three trophic levels of 12.8 mg.L⁻¹ ($12.8/1000 = 0.0128$ mg.L⁻¹).

However, TF obtained from chronic data remains a priority over TF obtained from acute data since considered as more representative of long-term effects and incorporating the principles of the Ecolabel CDV criterion and of regulatory environmental risk assessment.

Methodology relevance depending on the functional category

Depending on the functional category, defined by service provided to the consumer, an improvement of a formula's environmental profile can be challenging. A biodegradability average of the functional category already > 90% leaves little room for developing improved new formulae. For functional categories belonging to sunscreen products, UV filters can generally not be replaced even they are usually not readily biodegradable, because they are designed to be stable for efficient UV protection. The environmental impact of products from other functional categories is often difficult to improve through biodegradability and/or GWFE. In this case, improvements can be sought on other types of environmental impacts such as ingredient sourcing or product packaging (e.g. for make-up products).

Conclusion

Within the framework of its sustainable commitments, L'Oréal Group developed its own ecodesign methodology to reduce the environmental impact of the new cosmetic formulae marketed since 2014. This methodology focuses on three strategic indicators of rinse-off products: biodegradability, GWFE (grey water footprint adapted for ecodesign) and a global indicator complementary to these two specific indicators. These indicators are determined from environmental data on ingredients in the formula.

Data availability and data quality on cosmetic ingredients are essential for a good comparison and an improvement of the environmental impact of formulae. Data update remains the most important issue to solve since automatic update is not feasible yet and only expert judgement can address this update today.

The application of three ecodesign indicators does not systematically cover the overall potential impacts on the environment but the simplification introduced by a limited number of indicators is essential to engage formula developers to efficiently apply the ecodesign methodology and embed it in their development process.

By applying these easy-to-use methodologies, highly biodegradable products ($\geq 95\%$ based on ingredient composition) have been developed and marketed [38], with a low GWFE. This new ecodesign approach shows that embedding sustainability in developing new cosmetic products can also open up new innovative opportunities [39].

In addition to the improvement of biodegradability and GWFE of new cosmetic formulae, the present ecodesign strategy integrates other sustainable actions on the whole life cycle of cosmetic products. The use of ingredients sustainably sourced and/or derived from green chemistry and the improvement in the environmental profile of packaging make also part of this strategy.

Nowadays, all new cosmetic products of L'Oréal Group are therefore developed to reach the highest standards in terms of efficiency, cosmeticity and sustainability. In parallel, the Group is developing LCA-based approaches encompassing the impact of

products throughout their life cycle in accordance with the principles of the European Product Environmental Footprint project (PEF) [40].

Acknowledgement

The author wants to thank Pascale Cothias for her valuable inputs on ecodesign of new shampoo formulae (L'Oréal) and Sacha Laruelle (CEHTRA consulting) for external data gathering. All authors are L'Oréal employees.

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