# SOLUBLE FILMS IN SINGLE-DOSE DETERGENT PRODUCTS

# Information on their purpose, technical characteristics, testing and usage

Provided by A.I.S.E. in support of the study for the European Commission on "Microplastics pollution – measures to reduce its impact on the environment"

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## Introduction & executive summary

This document is provided by A.I.S.E. in the context of the Commission's study "Microplastics pollution – measures to reduce its impact on the environment", to answer questions raised by the consultants (UMons and BIOIS) in relation to the use of soluble films in detergent capsules. The latter was identified as an additional source for investigation in this study in February 2022, and this document follows up on bilateral discussions between A.I.S.E. experts and the consultants by providing further information on the purpose and role of soluble films in detergent capsule products, technical properties of the films, the basis for their biodegradability testing and sales volumes (presumed to equate approximately to volumes released in use).

A.I.S.E. members are willing to supplement this information with their own evidence (e.g. test reports) under conditions of confidentiality, given the proprietary and commercially sensitive nature of the data.

This document provides information elaborating on the following points:

- Soluble films based on polyvinyl alcohol (PVOH) are used to enable the practical and environmental benefits of unit dose detergent products for laundry and automatic dishwashing (and some other products to a much lesser extent).
- PVOH films do not meet the commonly-understood EU definition of microplastics.
- Different grades of PVOH have different behaviour in the environment.
- The use of PVOH film in liquid laundry detergent capsules and automatic dishwashing products does not lead to accumulation in the environment, since the products are soluble and biodegradable.
- OECD 301 is an appropriate screening test to demonstrate biodegradation that is widely used in industry and recognised by authorities.
- Estimates of PVOH release in Europe can be provided based on actual retail sales data combined with assumptions relating to product composition.





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# List of abbreviations

ADW	Automatic dishwasher/dishwashing					
A.I.S.E.	International Association for Soaps, Detergents and Maintenance Products (Association Internationale de la Savonnerie, de la Détergence et des Produits d'Entretien)					
ASTM	American Society for Testing and Materials					
BOD	Biological Oxygen Demand					
CLP	Classification, Labelling and Packaging of substances and mixtures (Regulation (EC) 1272/2008)					
(th)CO <sub>2</sub>	(Theoretical) Carbon dioxide					
DH	Degree of hydrolysis					
DOC	Dissolved Organic Carbon					
DP	Degree of polymerisation					
ECHA	European Chemicals Agency					
(US-)EPA	(United States) Environmental Protection Agency					
F/M	Food-to-Microorganism (ratio)					
LLDC	Liquid laundry detergent capsule					
(th)O <sub>2</sub>	(Theoretical) Oxygen					
OECD	Organisation for Economic Cooperation and Development					
PBT	Persistent, Bioaccumulative and Toxic					
PSP	Product Stewardship Programme (of A.I.S.E.)					
PVAc	Polyvinyl acetate					
PVOH	Polyvinyl alcohol					
WWTP	Wastewater treatment plant					



## 1. Why soluble films are used

Cleaning products such as laundry detergents and dishwashing products hold an important place in the hygiene routines of households around the world and have done so for a very long time. Detergents are constantly evolving to set new performance levels of cleaning and energy efficiency and to make use of new opportunities opened up by novel chemistries as well as in response to regulatory developments. After free-flowing dry detergents and liquid detergents, one of the more recent forms emerging is unit dose laundry products. Since their introduction in the early 2000's these products have seen a steady growth, attributed to their convenience for the consumer who appreciates the simplification of doing the laundry with the "less mess" and "right dose" attributes which this form offers.

Liquid laundry detergent capsules (LLDC's) have several characteristics which make them stand out from the traditional laundry forms. The volume of each LLDC is considerably lower than that of an equivalent dose of traditional detergent, yet cleans the same full load of laundry. All active ingredients are contained in a single unit dose capsule, which dissolves after contact with water inside the washing machine and then releases the detergent. In addition to this convenience, the high level of concentration leads to lower product amounts/job needed to be transported through the supply chain, which is a sustainability advantage by reducing CO<sub>2</sub> emissions. Additionally, the film performs the function of containment hence enabling the use of high-efficiency cleaning agents that facilitate low-temperature wash cycles and low-water wash cycles (which directly improves on sustainability, as a significant portion of the carbon emissions from a laundry cycle comes from the use phase of heating water¹).

As a unit dose, precise portion control is a key feature to dispense only the quantity needed per load, effectively reducing excessive use and consumer/end user overdosing. With LLDC all of the detergent ends up in the wash, with no residues left over to be disposed of in traditional packaging (or rinsed down the sink). While this example talks about liquid detergent unit dose, a parallel argument can be made for automatic dishwashing detergent unit dose products which have also seen a shift from free-flowing forms to unit dose executions.

The key enabler of LLDC is the capsule material, a water-soluble film, which holds the concentrated detergent solution. This film is generally based on PVOH, with polymer backbone modifications and specific performance additives. The films are developed such that they readily dissolve as intended in the washing process, including in sustainability driven cold water cycles, as well as to meet the technical challenges that mass volume production and regulatory compliance create. Frequently the product contains multiple compartments, allowing the formulators to separate ingredients which at the elevated concentrations of LLDC would not be compatible with each other.

To ensure consumer safety, and avoid spillage, the film is designed not to dissolve and rupture during routine transport and handling (e. g. when touched with wet hands); to resist compression (e. g. during transport or dosing); and to trigger an aversive reaction in case of oral contact. These features are required for liquid laundry detergent capsules in the EU under Regulation (EU) No. 1297/2014 (amending the CLP Regulation (EC) 1272/2008) which was put in place to help reduce accidental exposures involving young children observed in the market after introduction of the

<sup>&</sup>lt;sup>1</sup> Jay S. Golden, Vairavan Subramanian, Gustavo Marco Antonio Ugarte Irizarri, Philip White & Frank Meier (2010) Energy and carbon impact from residential laundry in the United States, Journal of Integrative Environmental Sciences, 7:1, 53-73



form. These successful risk management steps have been reapplied in most markets around the world as regulations or as voluntary programmes (A.I.S.E. PSP<sup>2</sup>, ASTM<sup>3</sup>).

The volumes of LLDC PVOH used have increased in line with the growing popularity of the LLDC form and, while for the EU market the film has been deemed a packaging item, it has nevertheless been designed to biodegrade like the rest of the LLDC ingredients as it travels down the wastewater stream. The PVOH grade used for cleaning products - LLDCs as well as automatic dishwashing products - is specially designed to fulfil the seemingly contradictory needs of dissolution, technical barrier characteristics needed for product stability, robustness and biodegradation.

### What is the definition of a microplastic?

The definition of microplastic has been extensively assessed and the European Chemicals Agency defines a microplastic as particles containing solid polymer, to which additives or other substances may have been added, and where  $\geq 1\%$  w/w of particles have (i) all dimensions [1nm  $\leq x$ ]  $\leq 5$  mm, or (ii) a length of [3 nm  $\leq x$ ]  $\leq 15$  mm and length to diameter ratio of >3. . [this term] shall not apply to polymers that are (bio)degradable . . . with a water solubility > 2 g/L.  $^4$ 

The proposed REACH restriction on intentionally-added microplastics is a near-final legislation of broad scope, which implicates microplastic polymers that are defined as "solid particles" below 5 mm in commerce at concentrations above 0.01% by weight. Derogations from the restriction include materials that are water soluble (>2 g/L) and biodegradable as determined by a series of OECD biodegradation screening tests, which are broadly applied and are used to evaluate surfactants for regulatory acceptance. ECHA guidance to evaluate these properties includes test standards such as OECD 105 or 120 for water solubility and OECD 301B, C, D, F or OECD 302B as screening approaches for biodegradability.

### Does PVOH meet ECHA's definition of a microplastic?

The film used in liquid laundry detergent capsules (LLDC's) and automatic dishwashing (ADW) capsules is larger than 5 mm (40 mm x 40 mm represents average size of PVOH resin/film). The film is also demonstrated to be soluble in water at >2 g/L at 21°C following OECD 120. Furthermore, PVOH is biodegradable as evidenced by meeting the pass criteria for stringent OECD test guidelines adopted by ECHA to evaluate persistence (see chapters 2 and 3 of this document for more information). According to this definition the water-soluble film used for liquid detergent capsules is not a microplastic. This is because the film size is outside of the microplastic range, and the film is both water-soluble and biodegradable.

<sup>&</sup>lt;sup>2</sup> A.I.S.E. <u>Product Stewardship Programme on liquid detergent capsules</u>

<sup>&</sup>lt;sup>3</sup> ASTM standard F3159-15, Consumer Safety Specification for Liquid Laundry Packets

<sup>&</sup>lt;sup>4</sup> Opinion on an Annex XV dossier proposing restrictions on intentionally-added microplastics, ECHA RAC/SEAC, 10 December 2020

## 2. Technical properties of polyvinyl alcohol films

Polyvinyl alcohol (PVOH) is a white, non-toxic, semi-crystalline polymer synthesized by polymerization of vinyl acetate to first polyvinyl acetate (PVAc) and further converting a specific portion of the acetate groups to alcohol groups (called saponification or hydrolysis) to get PVOH.<sup>5,6</sup> Consequently, the degree of hydrolysis (DH) is the percentage of acetate groups that have been converted to alcohol groups. Another key element is the length of the polymer quantified as degree of polymerization (DP). The DH and DP are key in determining physical properties, performance in application, and solubility (temperature and time required for dissolution).

Typical grades of commercialized PVOH available today range from 77 – 99% DH, with 77 - 90% DH considered to be partially hydrolysed and above 90% fully hydrolysed.<sup>7</sup> PVOH with DH values from 85 – ~90% are considered to be cold water soluble and will achieve full solubility in water colder than 20°C, and obviously also at warmer temperatures. These grades of PVOH are most commonly used in the films used for liquid laundry detergent capsules (LLDC) and automatic dishwasher (ADW) capsules, and are specifically selected by design to have the best solubility over a wide spectrum of washing conditions (cold water, low-water, etc.) backed by accredited solubility testing such as OECD 120.

High-DH grades of PVOH are not used in LLDC or ADW applications where solubility is a requisite, but rather in applications that use the other exceptional aspects of PVOH such as barrier properties, ductility, oil-resistance, non-toxicity, etc. These uses include food packaging, fibres for fishing nets, textile coatings, etc.<sup>8</sup> and account for over 95% of the PVOH usage in the world by volume.

The PVOH film used for making detergent capsules/packets is designed to dissolve completely within a matter of minutes (depending on factors such as water temperature, agitation, time, etc.) in the machine-assisted cleaning process (washing machine or dishwasher). In this typical laundry machine or automatic dishwasher cycle the PVOH is dissolved and is "broken down" to the molecular level. The detergent capsule/packet (made of PVOH film encapsulating the detergent) has less than 1 g of PVOH which is dissolved in approximately 40-85 litres of water during the wash cycle. At the end of the cycle, the wash water is then expunged/drained into water-treatment facilities where the PVOH is biodegradable.

PVOH is recognized as one of the very few water-soluble synthetic polymers susceptible to ultimate biodegradation<sup>9</sup>. This is confirmed by means of ready biodegradation screening tests (OECD 301) across a range of films used in detergent applications<sup>10</sup>. Accreditation agencies in the EU such as Ecolabel and Nordic Swan, and the US-EPA's Safer Choice program, utilise these very tests (OECD301) as criteria for evaluating biodegradation and subsequent approval and certification for LLDC and ADW products as needed.

<sup>&</sup>lt;sup>5</sup> K. Noro. *Br. Polym. J.* 2, 128 (1970).

<sup>&</sup>lt;sup>6</sup> Y. Sakaguchi; Z. Sawada; M. Koizumi; K Tamaki. Kobunshi Kagaku 23, 890 (1966).

<sup>&</sup>lt;sup>7</sup> C. A. Finch. *Polyvinyl Alcohol Properties and Applications*, Chapter 2, 17 (1973).

<sup>8</sup> https://www.kuraray-poval.com/applications/

<sup>&</sup>lt;sup>9</sup> Chiellini, E.; Corti, A.; D'Antone, S.; Solaro, R. *Biodegradation of poly (vinyl alcohol) based materials*. Prog. Polym. Sci. 2003, 28, 963–1014

<sup>&</sup>lt;sup>10</sup> D. Byrne, G. Boeije, I. Croft, G. Hüttmann, G. Luijkx, F. Meier, Y. Parulekar, G. Stijntjes, *Tenside Surfactants Deterg.* 2021, 58, 88.



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A common question about these reported results (and tests) is whether they really replicate the actual behaviour of a biodegradable material in real-world conditions such as wastewater treatment plants (WWTP). The OECD 301 series of biodegradation tests (see chapter 3 of this document) are considered stringent screening tests, conducted under aerobic conditions, in which a high concentration of the test substance (in the range of 2 to 100 mg/L) is used and ultimate biodegradation is measured by non-specific parameters like Dissolved Organic Carbon (DOC), Biochemical Oxygen Demand (BOD) and CO<sub>2</sub> production. These tests are considered stress tests for the test chemical since the system does not have an environmentally realistic ratio of test chemical to microbes (which in an actual WWTP would be orders of magnitude higher). The inoculum is sourced from a well operated domestic wastewater treatment plant that has a diverse and robust microbial population, and no pre-exposure to the test chemical is allowed. In these studies, the test material is sole carbon and energy source for the population of microorganisms to use and grow. The high test-substance concentration creates a risk for toxicity for the microorganisms, and it is generally accepted by regulators that a positive result in a test for ready biodegradability can be considered indicative of rapid and ultimate degradation in most environments including wastewater treatment plants.

Even though WWTPs are the obvious and expected environmental compartments for end-of-life for PVOH based on correct usage/disposal, it is indeed necessary to ensure biodegradation<sup>11</sup> for PVOH films in other compartments such as soil, river water, etc. Extensive publications and reports exist on such studies (Matsumura has detailed a lot of this here<sup>12</sup>) with the first degradation in soil<sup>13</sup> reported back in the 1930s. In comparison to WWTP sewage systems, the biodegradation rates in soil and marine environments are expectedly slower yet still indicative of ultimately biodegrading as per established mechanisms. Additionally microbial organisms that are already acclimatized to degrading PVOH exist in WWTPs and septic systems as a consequence of generations of use of PVOH in paper and textile coatings, thus disavowing the need for acclimation time or lag phases.

As seen from the above section, it is clear that the film is a very key component in the process to ensure safe delivery of these super-compacted cleaning agents to the consumer. The current films for LLDCs have been developed and optimized over decades of research on not only PVOH chemistries but other water-soluble systems. PVOH has unique attributes that qualify it in the ideal overlapping area of performance, safety and sustainability. Due to the ability to design the PVOH molecule (DH, DP, etc.), it is possible to make it compatible with a range of cleaning chemistries and harsh chemicals. This is a very important need for consumer safety, because if the LLDCs show any adverse interaction then there is a possibility of accidental exposure for the user or potentially even creating insoluble LLDCs. PVOH by design is completely water-soluble (tested as per OECD 120) and not just water dispersible as this may lead to residue issues on textiles/dishes. Dispersions may also lead to being vectors for bioaccumulation in the wastewater stream for other toxic chemicals. It is proven to be non-toxic and non-inhibitory to aquatic

https://www.kuraray-poval.com/fileadmin/technical\_information/brochures/poval/agriculture-brochure-kuraray-poval-web.pdf

<sup>&</sup>lt;sup>12</sup> Shuichi Matsumura, Biodegradation of Poly(vinyl alcohol) and its Copolymers. 2002, https://doi.org/10.1002/3527600035.bpol9015

<sup>&</sup>lt;sup>13</sup> Nord, F. F. (1936) Dehydrogenation ability of Fusarium lini B, Naturwissenschaften, 24, 793



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life and organisms, as seen from various published reports<sup>14,15</sup>. A very important aspect to be noted is the property of PVOH films to impart a high degree of structural integrity to the LLDCs: for safety reasons, LLDCs need to have a minimum seal strength and compressive strength (as defined by Regulation (EU) 1297/2014). This ensures that the contents are not accidentally expelled.

We as an industry are obviously always innovating and developing solutions that may present a different aspect on sustainability or other properties coming from alternative materials. But it should be clear that such innovation is the natural next evolutionary step and not driven by any necessity to overcome any shortcomings or problems with the incumbent material. Decades of diligent scientific research and development lie behind this product innovation, which has been built on transparency, accountability and stewardship.

<sup>&</sup>lt;sup>14</sup> Biodegradation of Polyvinyl Alcohol, R.J. Axelrod and J.H. Phillips (Air Products and Chemicals Inc.), presented at the Plastics Waste Management Workshop, New Orleans, LA, 9-12 December 1991

<sup>&</sup>lt;sup>15</sup> Pecquet, A., McAvoy, D., Pittinger, C. and Stanton, K. (2019), Polymers Used in US Household Cleaning Products: Assessment of Data Availability for Ecological Risk Assessment. Integr. Environ. Assess. Manag., 15: 621-632. <a href="https://doi.org/10.1002/jeam.4150">https://doi.org/10.1002/jeam.4150</a>



# 3. Basis for OECD 301 tests and threshold level of 60%

### **Background**

Industrialized nations recognized the need to assess the biodegradability of substances entering the environment in the 1950s and 1960s. Early regulatory initiatives generated laboratory work sponsored by industry and academia, which in turn led to numerous clean water regulations and the associated industrial response.

As biodegradability became an increasingly important design criterion for consumer product R&D groups, the need for a rapid screening test emerged. Pioneering work in this field was conducted by, among others, R.N. Sturm<sup>16</sup> in 1973.

In the years following Sturm's publication, numerous investigators expanded on, and refined Sturm's work<sup>17</sup> eventually resulting in a set of six tests adopted by the Organisation for Economic Cooperation and Development (OECD) in July of 1992<sup>18</sup>. Since that time, these tests have become international standards for assessing the biodegradability of materials introduced into our waterways. Over the past several decades, these OECD standards have guided laboratories in the assessment of the biodegradability of a wide range of chemical substances.

These OECD screening tests are utilized in part of the tiered approach by ECHA and US-EPA to broadly evaluate and identify persistency of chemicals under PBT classification schemes, such as surfactants and other organic chemistries and extending now to microplastics.

### **OECD301 Biodegradability Tests**

There are six tests in OECD 301, and each method results in an assessment of aquatic effluent biodegradability in fully aerated conditions. Briefly, OECD 301A measures the disappearance of organic carbon, OECD 301B quantifies the generation of carbon dioxide, OECD 301C, 301D, and 301F monitor oxygen uptake, and OECD 301E monitors the disappearance of dissolved organic carbon<sup>19</sup>.

These tests are stringent tests that provide limited opportunity for biodegradation, the idea being that a compound giving a positive result in such a test may be assumed to biodegrade quickly in municipal wastewater treatment plants<sup>20</sup> and the environment<sup>21</sup>. These biodegradability tests rely on the use of heterotrophic bacteria under controlled conditions to consume the organic substance being evaluated. Heterotrophs consume carbon for two reasons - for respiration and for biomass development (cellular growth).

<sup>&</sup>lt;sup>16</sup> R.N. Sturm, "Biodegradability of Nonionic Surfactants: Screening Test for Predicting Rate and Ultimate Biodegradation", Journal of American Oil Chemists Society, 1973.

<sup>&</sup>lt;sup>17</sup> No <sup>2</sup> ENV Monograph 98: "Detailed Review Paper on Biodegradability Testing". Paris 1995.

<sup>&</sup>lt;sup>18</sup> OECD Guideline for Testing of Chemicals Adopted by the Council on July 17, 1992: "Ready Biodegradability".

<sup>&</sup>lt;sup>19</sup> "Summary of OECD 301B Biodegradability Testing and Classifications", Respirtek Consulting Laboratory, June 23, 2021.

<sup>&</sup>lt;sup>20</sup> Struijs, J. Stoltenkamp, J., Van de Meent, D., 1991. A spreadsheet based model to predict the fate of xenobiotics in a municipal wastewater treatment plant. Wat. Res. 25:891-900.

<sup>&</sup>lt;sup>21</sup> De Bruijn, J., Struijs, J., 1997. Biodegradation in chemical substances policy. In: Hales, S.G., Feijtel, T., King, H., Fox, K., Verstraete, W. (Eds.), Biodegradation Kinetics: Generation and Use of Data for Regulatory Decision Making, SETAC-Europe Workshop, 4-6 September 1996, Port-Sunlight, UK. SETAC-press, Brussels, Belgium, pp. 33-46.



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These studies are considered conservative stress tests as they employ high test chemical concentrations and low levels of inocula that are not realistic when compared to actual environmental conditions<sup>22,23</sup>. The low levels of inocula limit the microbial diversity which is unrealistic compared to actual environmental compartments<sup>21,24</sup> and time is needed for the microbial population to adapt to the test chemical as the sole food source to use and grow to rapidly degrade the high concentration of test chemical.<sup>25,26,27</sup> Finally, the low level of inocula used in the test significantly limits the microbial diversity when compared to actual environmental conditions.<sup>21,22</sup> The inoculum is sourced from a well operated domestic wastewater treatment plant that has a diverse and robust microbial population, and no pre-exposure to the test chemical is allowed.

In each of the respirometric tests, the OECD has determined that CO<sub>2</sub> generation amounting to 60% of the substrate organic carbon can model the complete consumption of the substrate, with the balance of the carbon (40%) being used for biomass creation.

#### 60% Pass Level indicates Total Utilisation

OECD defines ultimate biodegradation results at 60% as "The level of degradation achieved when the test compound is totally utilised by micro-organisms resulting in the production of carbon dioxide, water, mineral salts and new microbial cellular constituents (biomass)"<sup>18</sup>. The 60 percent threshold is not intended to mean that only 60% of the test substrate degrades <sup>16,19,28</sup>.

Theoretical calculations cited in Metcalf and Eddy<sup>29</sup> show that heterotrophs will use organic carbon from their food at a 42/58 ratio when biodegrading organic compounds consisting of carbon, hydrogen and oxygen. That is, for each 100 grams of organic carbon in the substrate in a biodegradation test, a maximum of 42 grams will end up as cellular biomass, and a minimum of 58% will convert to carbon dioxide as a result of cellular respiration.

In the OECD 301/310 screening tests, the test chemical is the only food source present for the microbial community. The growth yield (amount of carbon used for energy versus the amount of carbon converted to biomass) changes depending on the test chemical and the competent degrader. A typical growth yield is 40 to 50% indicating that 50-60% of the carbon is converted to carbon dioxide and 40 to 50% is incorporated into new biomass. The pass criterion for an

<sup>&</sup>lt;sup>22</sup> Thouand, G., Capdeville, B., Block, J., 1996. Preadapted inocula for limiting the risk of errors in biodegradability tests. Ecotoxicol. Environ. Saf. 33, 261-267.

<sup>&</sup>lt;sup>23</sup> Goodhead, A.K., Head, I.M., Snape, J.R., Davenport, R.J., 2014. Standard inocula preparations reduce the bacterial diversity and reliability of regulatory biodegradation tests. Environ. Sci. Pollut. Res. 21, 9511-9521.

<sup>&</sup>lt;sup>24</sup> Struijs, J., van den Berg, R., 1995. Standardized biodegradability tests: extrapolation to aerobic environments. Water Res. 29, 255-262.

<sup>&</sup>lt;sup>25</sup> van Ginkel, C.G., Haan, A., Luijten, M.L.G.C., Stroo, C.A., 1995. Influence of the size and source of the inoculum on biodegradation curves in closed-bottle tests. Ecotoxicol. Environ. Saf. 31, 218-223.

<sup>&</sup>lt;sup>26</sup> Itrich, N.R., McDonough, K.M., Van Ginkel, C.G., Bisinger, E.C., Lepage, J.N., Schaefer, E.C., Menzies, J.Z., Casteel, K.D., Federle, T.W., 2015. Widespread microbial adaptation to I-Glutamate-N,N-diacetate (L-GLDA) following its market introduction in a consumer cleaning product. Environ. Sci. Technol. 49, 13314-13321.

<sup>&</sup>lt;sup>27</sup> Francois, B., Armand, M., Marie-Jose, D., Thouand, G., 2016. From laboratory to environmental conditions: a new approach for chemical's biodegradability assessment. Environ. Sci. Pollut. Res. 23, 18684-18693.

<sup>&</sup>lt;sup>28</sup> D. Weytjens, I. Van Ginneken, HA Painter, "The Recovery of Carbon Dioxide in the Sturm Test for Ready Biodegradability", Chemosphere, 28, 1994, 801-812.

<sup>&</sup>lt;sup>29</sup> Metcalf and Eddy Inc. Wastewater Engineering: Treatment and Resource Recovery. Boston, McGraw Hill, 2003.



OECD 301/310 screening test was set at 60% thCO<sub>2</sub> evolution or thO<sub>2</sub> consumption based on knowledge of these typical growth yields.

The 60% threshold is based on the Food-to-Microorganism (F/M) ratio and an inoculum source that is low in residual carbon. High F/M ratios like those in the OECD biodegradability tests are expected to produce an assimilation percentage near the theoretical value<sup>30</sup>, which is the fundamental basis for the 60% conversion of the organic carbon to CO<sub>2</sub> as the criterion from which one can assume complete biodegradation in the aquatic environment<sup>18,19,31</sup>.

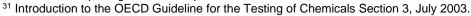
As to the correlation of the test relative to the residence time typical of wastewater treatment facilities, these factors are scaled against the various concentrations of microorganisms and the lower F/M ratio that would be present in a real-world scenario. The total solids concentration in the OECD 301B test is 30 mg/L whereas in an activated sludge treatment facility, solids concentrations can be maintained between 3,500-5,000 mg/L. The lower inoculum concentration in the OECD test provides low CO<sub>2</sub> production in blank vessels without external addition of substrate (food source) and thus a high signal-to-noise ratio in the quantification of biodegradability extent. The OECD literature clearly indicates that a "passing" result, according to the specific test (>60% for 301B), can be expected to translate accordingly into the aquatic environments. Extrapolation of 60% biodegradation to a higher percentage to estimate a time to complete mineralization would be based on a misconception of the pathways for carbon in microbial metabolism<sup>19</sup>.

### **Summary**

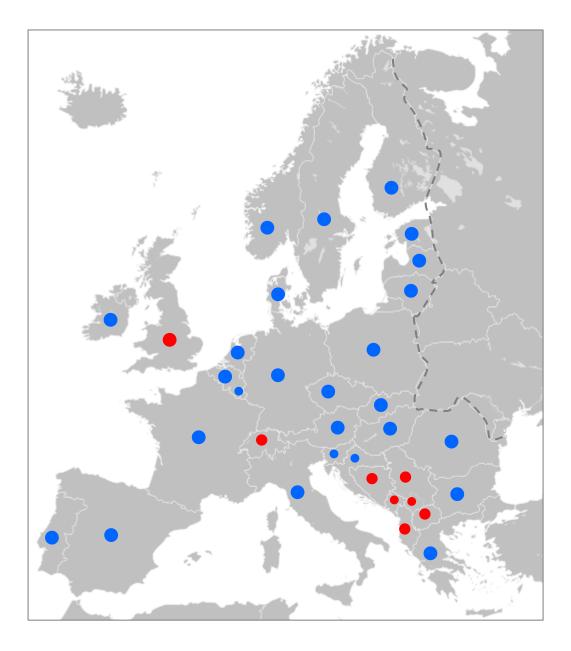
The screening test that Sturm et al created in the 1970s and 1980s was adopted by OECD in 1992 with the F/M refinement discussed above. As with any significant test method, this adoption was accompanied by peer reviews, complementary laboratory work, and a discussion of alternative tests.

In recognition of this process, OECD sponsored an additional Environment Monograph, #98: "Detailed Review Paper on Biodegradability Testing", Paris, 1995<sup>17</sup>. In this paper they conclude that, after an extensive global literature review, the procedures outlined in OECD 301 did not need to be altered. To this day OECD 301, including the current pass/fail threshold, remains the industry standard in biodegradability assessment. Further, this guideline is used by ECHA to evaluate persistence of a broad range of chemicals under PBT schemes and more recently in the proposed derogation for microplastics under REACH.

<sup>&</sup>lt;sup>30</sup> Marcos van Sperling, Basic Principles of Wastewater Treatment, Volume 2, IWA Publishing, New York, 2007.



# 4. Information on market volumes



Colour key to countries covered in tables below for Europe geographic area

Retail sales data from Euromonitor International, © 2022



### Liquid laundry detergent capsules – retail volume (in kilotonnes)

	2017	2018	2019	2020	2021
EU + NO	103.2	121.1	137.3	152.6	167.1
Other Europe	55.7	60.7	64.4	66.4	69.3
TOTAL	158.9	181.8	201.7	219.0	236.4
PVOH release (estimated)*	7.9	9.1	10.1	10.9	11.8

<sup>\*</sup> Based on conservative assumption of max. 1g PVOH film per capsule (in range 13 – 28g, assumed average 20g), i.e. **5%**.

Members report that these figures may be slightly underestimated based on their own PVOH consumption (which may also include internal wastage), but no better basis is currently available to calculate quantities of film placed on the market/released by dissolution in use.

### Automatic dishwashing tablets - retail volume (in kilotonnes)

NB. Euromonitor provides sales data for <u>all</u> tablets without differentiation between wrapped/unwrapped or removable/soluble film.

	2017	2018	2019	2020	2021
EU + NO	197.5	202.0	209.2	237.3	242.9
Other Europe	42.9	43.3	44.8	50.1	51.8
TOTAL	240.4	245.3	254.0	287.4	294.7
Total using soluble film <sup>†</sup>					
PVOH release (estimated)**					

<sup>&</sup>lt;sup>†</sup> To be populated when reliable source identified for estimate

### **WC** care

PVOH soluble film is also used in a small proportion of toilet cleaning products such as rim hangers and cistern blocks.

Estimated total PVOH quantity: ~0.03 kt/annum (2021).



<sup>\*\*</sup> Based on typical 1% PVOH film per tablet (i.e. 0.20 g in a 20 g tablet).