Biodegradability of Water-Soluble and Water-Dispersible Polymers in Respirometric Laboratory Methods

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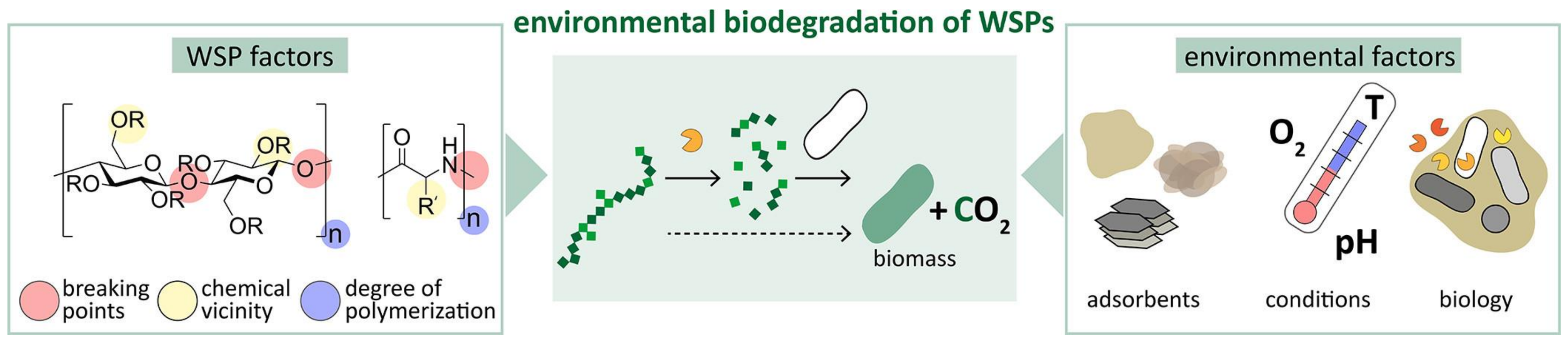
Water-soluble and water-dispersible polymers can be found in a large range of applications in our everyday life, from cosmetics to detergents, from paints to agricultural formulations. For polymeric materials regulations world-wide are rapidly developing with greater focus on the assessment of the degradability of polymers in application leading to environmental exposure. This has put an increasing emphasis on the need to identify, and critically evaluate testing guidelines for assessing biodegradability of for polymeric materials. Multiple biodegradation testing methods are available. While developed for discrete substances, these may in principal be used to assess biodegradability of soluble and poorly soluble polymeric materials. These methods range from screening tests such as the OECD test guidelines (e.g., 301/310) for biodegradation, or media-specific methods such as ISO (e.g., 14852) and ASTM test guidelines (e.g., 5988). These methods have some commonalities such as reliance on non-specific analyses (i.e., O_2 consumption, DOC and CO_2) and offer some level of flexibility regarding inoculum type and concentration and test substance dose level. Unfortunately, systematic investigations about their applicability to different types of polymers are lacking and limited publicly data are currently available. In view of the extremely dynamic regulatory atmosphere, these knowledge gaps need to be urgently filled. In this study we present a series of experiments performed in different laboratories across the globe according to OECD, ISO and ASTM methods to study biodegradability of representative water-soluble and water-dispersible polymers. A set of synthetic polymers and modified polysaccharides have been used as case studies. Standard methods and modifications thereof to improve method deficiencies, ranging from polymer, inoculum ratio, microbial concentration and effect of temperature will be presented.

Polymer biodegradation processes & test methods



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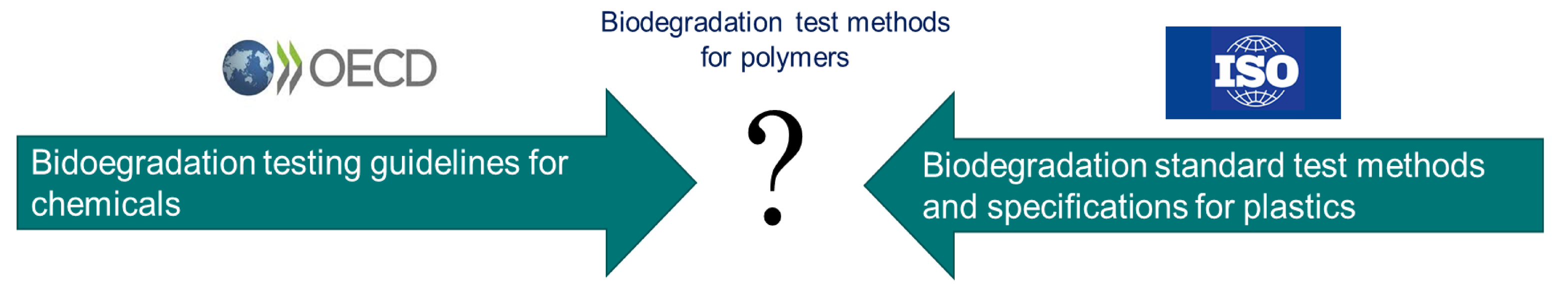


A.I.S.E. polymer biodegradation TF

Goals
- Assess methodology for (bio)degradation of soluble and poorly soluble polymers
- Work on test modifications and refinements and promote these tests for official recognition
- Provide data to inform regulators drafting guidelines for polymer persistence assessments

Engagement rules
- Each member of the working group must contribute by engaging in laboratory testing
- Testing can be performed in house or at contractor labs or even by academic cooperation partners

Case studies – phase 1

Test Material	Full name	Category	avg. Mw (g/mol)	Other characteristics (branching, DS, …)	Charge/reactive groups	Key Characteristics
MCC	Microcrystalline cellulose	Positive control	-	-	-	Non soluble polymer control, ISO test linkage
PEG 35000	Polyethylene glycol	Case study	35,000		Neutral	Mw
PVOH 18-88	Polyvinyl alcohol	Case study	130,000	DH = 88	Neutral	Mw, degree hydrolysis (DH)
CMC 0.6	Carboxymethyl cellulose	Case study	200,000	DS = 0.6	Anionic	Mw, degree of substitution (DS)
MGG	2,3-epoxypropyl-trimethylammonium modified guar gum	Case study	1,000,000-2,000,000	Homogenous charge density	Cationic	Mw, Charge density

Table 1: Polymers used as case studies for testing in phase 1 of polymer biodegradation taskforce

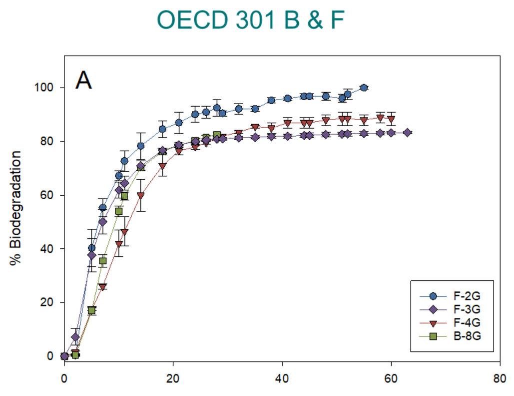
McDonough, Kathleen, et al. "Multi-laboratory evaluation of the reproducibility of polymer biodegradation assessments applying standardized and modified respirometry methods." *Science of The Total Environment* 901 (2023): 166339.

Abbreviation	Location	Test Method	Inoculum	Test substance concentration	Inoculum concentration
F-1G	CH	OECD 301 F	Sludge	100 mg/L	30 mg/L SS
F-1M	CH	OECD 301 F	Sludge	50 mg/L	30 mg/L SS
F-2G	DE (1)	OECD 301 F	Sludge	100 mg/L	30 mg/L SS
F-2M	DE (1)	OECD 301 F	Sludge	100 mg/L	120 mg/L SS
F-3G	US (1)	OECD 301 F	Sludge	50 mg/L ThOD	30 mg/L SS
F-3M	US (1)	OECD 301 F	Sludge	50 mg/L ThOD	60 mg/L SS
F-4G	NL (1)	OECD 301 F	Sludge	45-66 mg/L	30 mg/L SS
F-5M	NL (2)	OECD 301 F	Sludge	30-60 mg/L	35 mg/L SS
B-6M	US (2)	OECD 301 B	Sludge	40 mg C/L	60 mg/L SS
B-7M	DE (2)	ISO 14852	Sludge	130-300 mg C/L	120 mg/L SS
B-8G	US (3)	OECD 301 B	Sludge	12 mg C/L	17 mg/L SS
RW-8	US (3)	CO_2 Respirometry	River water	25-50 mg/L	865 mL river water

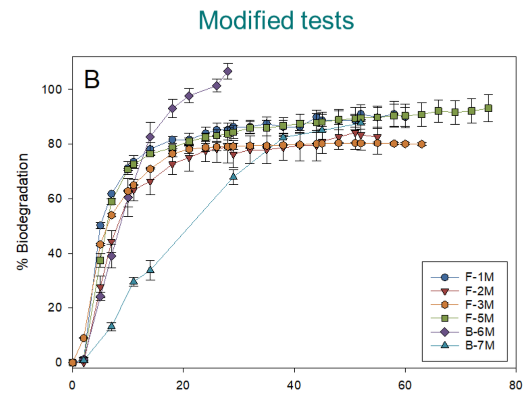
Table 2: Overview of experiments performed using activated sludge and river water as inoculum

MCC

OECD 301 B & F

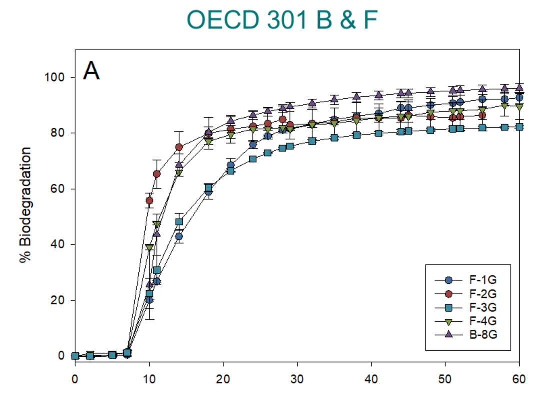


Modified tests



PEG 35000

OECD 301 B & F



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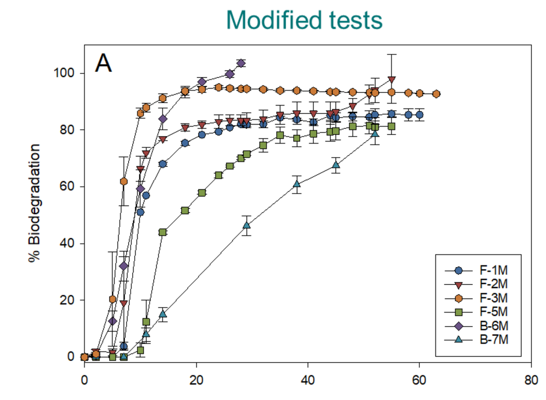
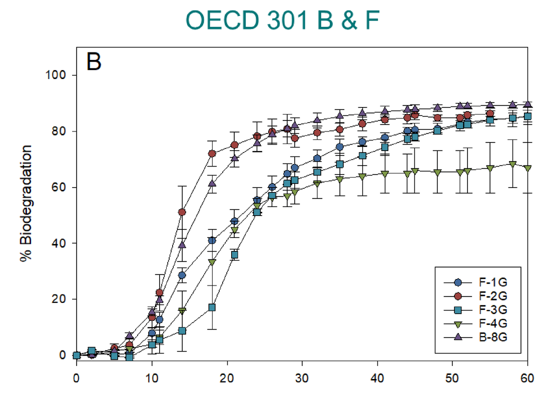
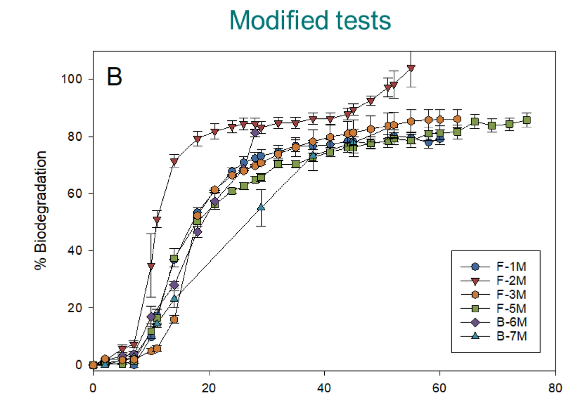


PVOH 18-88

OECD 301 B & F

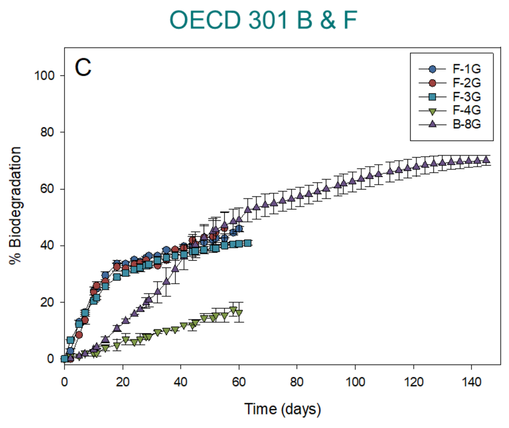


Modified tests

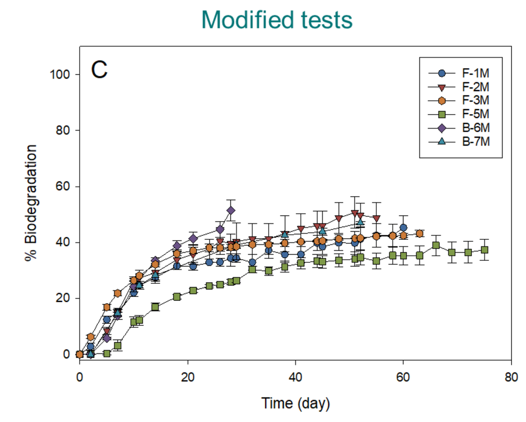


CMC 0.6

OECD 301 B & F

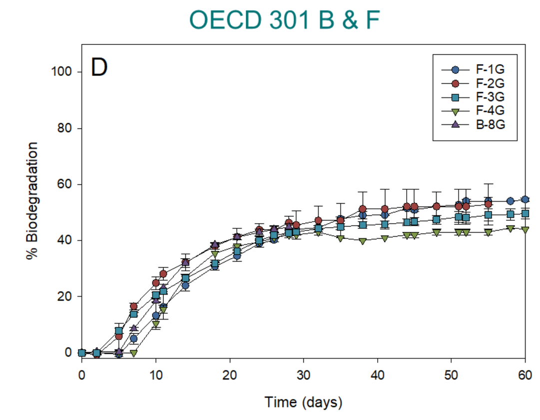


Modified tests

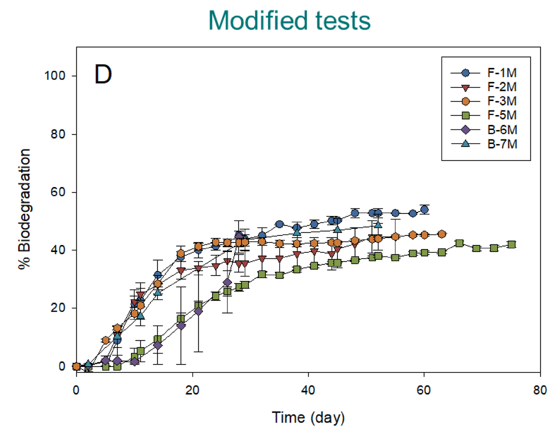


MGG

OECD 301 B & F

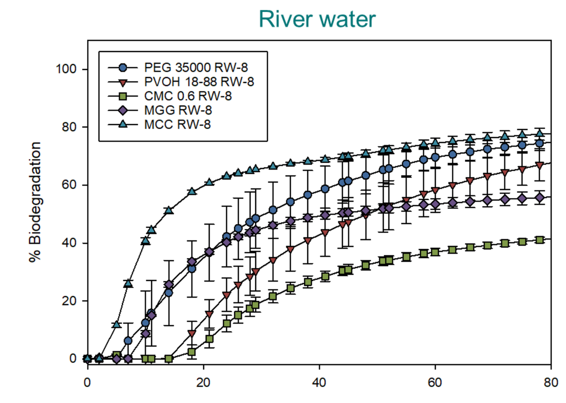


Modified tests

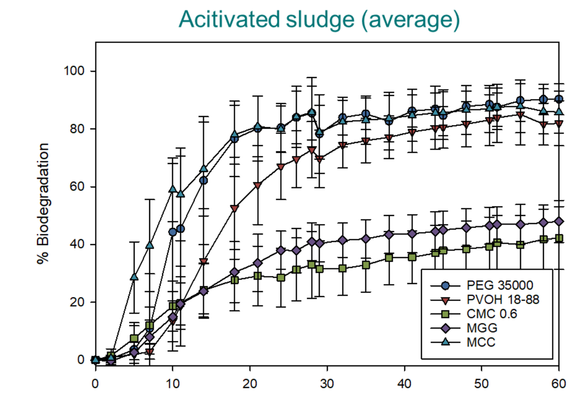


River water vs. activated sludge

River water



Acitivated sludge (average)



Conclusions and next steps

- High level of reproducibility across OECD 301 F & B, confirming reliability of the test method
- Modified studies did not show significant impact on extent of mineralization, making these tests a viable alternative to assess biodegradability of polymeric materials
- Similar extent of mineralization in studies with activated sludge and river water confirming environmental relevance of OECD 301 results
- Testing time extension required to reach full extent of mineralization. Modifications should be further explored when investigating polymeric materials
- Microcrystalline cellulose fulfils the criteria to be reference substance for biodegradation studies. Additional positive and negative controls to be identified
- Experiments ongoing to expand the data generated so far to other polymeric materials and guidelines and to explore new and modified test methods

If you are interested in collaborating with us, let us know