Biodegradability of Polymers in Aquatic Test Systems: ISO 14852 - Feasibility Study for Different Concentrations of Test Item and Inoculum

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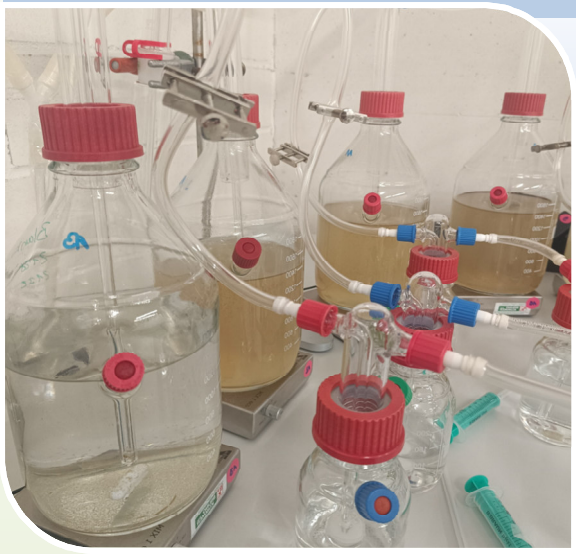
Introduction

Polymers are a product group that is increasingly becoming the focus in the topic of biodegradability and persistency. Due to their broad spectrum of properties, both synthetic and natural polymers play essential roles in everyday life. Guideline EN ISO 14852 [1] is one out of several to proof biodegradability of polymers in aquatic test systems. According to the *Official Journal of the European Union, Entry 78 [2]*, the EN ISO 14852 [1] is one of the permitted test methods, to demonstrate that the polymer is degradable (group 4, three compartments).

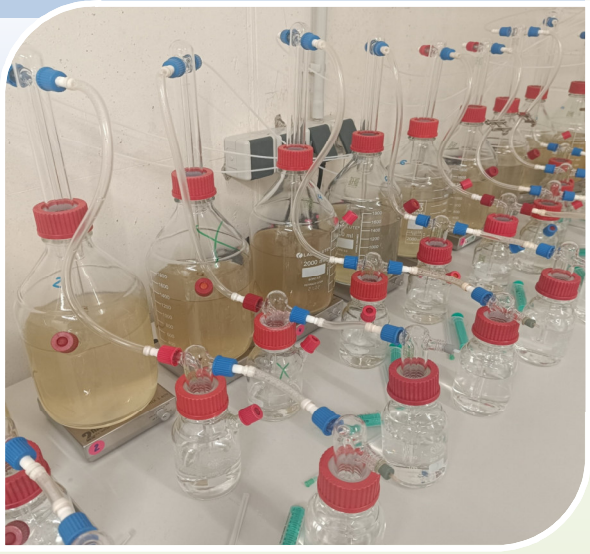
The ISO 14852 guideline [1] follows the OECD 301B guideline [3] very closely in terms of testing principles, but the permitted concentration range of the test concentration and inoculum concentration is much larger (test concentration: 30 - 2000 mg/L TOC (in comparison: OECD 301B: 10 – 20 mg/L TOC), inoculum concentration: 30 – 1000 mg/L dry solids (in comparison: OECD 301B: 30 mg/L dry solids)). Furthermore, according to the Official Journal of the EU [2], the test duration may be extended up to six months, this could offer an advantage in demonstrating the degradability of more complex substances, such as polymers, in an aqueous medium. According to a ring test mentioned in the ISO Guideline [1], e.g. CO_2 evolution has only been determined in the lower inoculum concentration range of 30 mg/L dry solids.

The feasibility test described in this study examines the technical feasibility of using various concentrations of test substance and inoculum in ISO 14852 (2021) [1].



Test design

Test item:	Sodium carboxyl methyl cellulose (**CMC 0.6**)
Test item concentration	30, 90 and 300 mg/L TOC
Inoculum concentration	30, 90 and 300 mg/L dry solid (d.s.)
Test duration	60 days
Test vessels:	2000 mL reactors, 250 mL Absorber flasks with NaOH as absorber medium for IC determination
Test volume	1500 mL
Reference item:	Cellulose, highly pure



Results:

Table 1: Degradation rates at different concentrations of test item and inoculum (test duration 60 days)

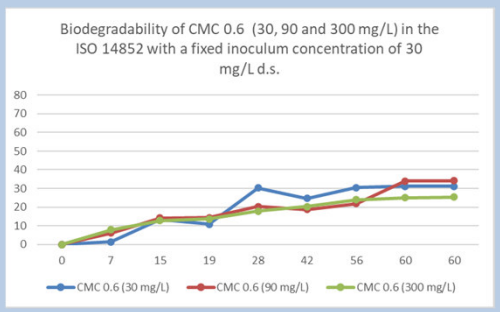
Inoculum concentration [mg/L d.s.]	Test item concentration [mg/L TOC] 30	90	300
30	31.1% (SD 9.2)	34.1% (SD 17.0)	25.3% (SD 7.2)
90	61.9% (SD 7.7)	68.0% (SD 5.9)	x
300	46.8% (SD 9.5)	x	23.1% (SD 1.1)

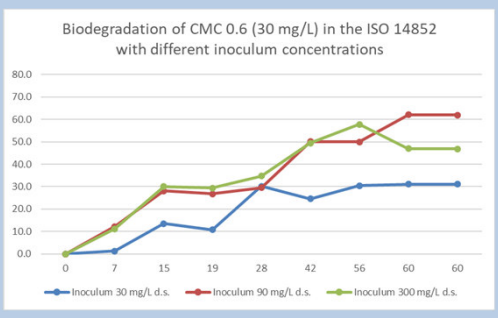
x = not determined

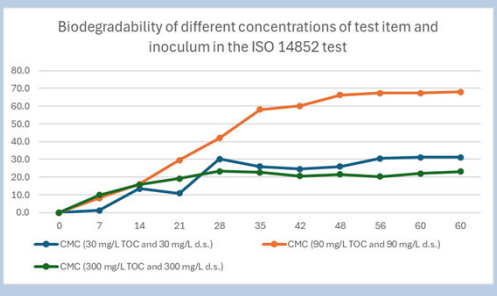
Table 2: CO_2 evolution in the blank controls with different inoculum concentrations (test duration 60 days)

Inoculum concentration	CO_2 evolution*
30 mg/L d.s.	48 mg/L
90 mg/L d.s.	99 - 110 mg/L
300 mg/L d.s.	247 - 310 mg/L

*Required CO_2 evolution in the OECD 301B is <70 mg/L within 28 days at inoculum concentration of 30 mg/L d.s.







Conclusions:

- Increase of test item concentration at constant inoculum concentration has no influence on the biodegradability of the CMC
- Increase of inoculum concentration leads to higher biodegradation extents of CMC up to a certain level. There appears to be an optimum, beyond which a further increase has a negative effect on the biodegradation
- CO_2 evolution in the blank controls increases in proportion to the rise in concentration. When scaled down to 30 mg/L, CO_2 evolution falls within the recommended range for Ready Tests (e.g. OECD 301B: < 40 mg/L))
- Test approaches with very high inoculum concentrations, such as 300 mg/L d.s. in this case (10 times higher than standard concentration of ready tests), lead to technical difficulties: CO_2 production is very high, meaning that the capacity of the absorber medium was insufficient. The detection of inorganic carbon (IC) in the TOC analyzer reaches its limits.

Outlook:

- Further tests with different test concentrations and different test substances should be performed to refine the test method
- The high inoculum concentrations caused technical difficulties in capturing and measuring the IC; further tests using a larger absorber volume or a change of absorber vessels within the test are required
- Further tests with longer test duration are required to monitor the development of the blank controls at higher inoculum concentrations and over a test period of up to six months.

[1] Guideline EN ISO 14852: *Determination of the complete aerobic biodegradability of plastic materials in an aqueous medium — Method using analysis of the released carbon dioxides (July 2021)*
[2] OECD Guideline for the Testing of Chemicals (July 1992) No. 301: *Ready Biodegradability, Method B: CO2 Evolution (Modified Sturm Test)*
[3] Official Journal of the European Union, Entry 78, Appendix 15 from 27 September 2023: Amending Annex XVII to Regulation (EC) No 1907/2006 of the European Parliament and of the Council concerning the Registration, Evaluation, Authorisation and Restriction of Chemicals (REACH) as regards synthetic polymer micorparticles

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