

Development of an extraction and analysis procedure for the quantitative evaluation of the content of MCPP and MCPP esters in polymer bitumen sealing membranes

Final report on the research project

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Summary

Polymer bitumen roof sealing membranes – such as those utilised for roof sealing purposes in green roofs – feature esters of 2- (4-chloro-2-methylphenoxy)-propionic acid (“mecoprop”, MCPP). When the mecoprop esters come into contact with water, they are washed out of the bitumen layer and mecoprop is released from the esters by hydrolysis. Through the radicide effect of mecoprop, polymer bitumen roof sealing membranes on green roofs are protected from root penetration. However, only the (R)-(+)-enantiomer “mecoprop-P”, which is mainly present in modern formulas for protection against root penetration, is effective.

An extraction procedure and analysis method have been developed and validated for MCPP esters to determine the content of MCPP and MCPP esters in roof sealing membranes (for homogeneity testing, for example). The extraction procedure involved punching out material samples from roof sealing membranes with a punch. The analytes were extracted by stirring in room-temperature toluene for 1 to 3 hours. To prepare the analytics, the extracts were filtered and diluted while changing the solvent. MCPP was analysed using UPLC-MS/MS. The MCPP esters (MCPP-P-2-ethylhexyl ester and MCPP-P-n-octyl ester) were determined using liquid injection GC-MS. Both the GC-MS analytics and the UPLC-MS/MS analytics highlight determination thresholds of below 1 µg/l, which corresponds to an area-specific content (m/m) of 0.1 g/m² of MCPP ester or 0.005 g/m² of MCPP.

The identification, detection and determination thresholds were determined to validate the GC-MS analytics and the overall procedure. Further elements that were determined include the linearity of the analysis procedure, the reproducibility of the calibration process, the recovery, the durability of the overall procedure, and the stability of the solutions. Tests were also carried out on extracts to assess the influence of the filtration process.

The validated method for determining the content of MCPP and its octyl esters (MCPP-P-2-ethylhexyl ester and MCPP-P-n-octyl ester) was applied to two polymer bitumen roof sealing membranes. The area-specific content of MCPP-P-n-octyl ester in roof sealing membrane 1 was 9.2 ± 0.5 g/m². Roof sealing membrane 2 contained 6.4 ± 1.1 g/m² MCPP-P-2-ethylhexyl ester. Traces of MCPP were found in both roof sealing membranes. The mole ratio (ester / carboxylic acid) was only 1.1 % for roof sealing membrane 2 and only 0.3 % for roof sealing membrane 1. The results clearly indicate that each ester is only hydrolysed on the surface or in the aqueous eluate – not in the bitumen roof sealing membrane itself.