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Section 2 **Test Methodology and Assessment**

Insight in moisture dynamics of wood treated with organosilicon compounds.

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Insight in moisture dynamics of wood treated with organosilicon compounds

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ABSTRACT

The SILEX project "Improving sustainability of construction materials using innovative Silicon based treatment" is a Life+ project with reference LIFE+11 ENV/BE/1046 and started in April 2013. This project intends to demonstrate that a new class of compounds can be used for wood treatment for an extended service life combined with enhanced new testing methodology. The project aims at demonstrating that treatments with silicon-based hydrophobers have a lower impact on the environment with a lower input of biocides.
This paper gives some results related to moisture dynamics and the relationship between simulated outdoor tests and laboratory methods.

Different initiatives related to service life prediction of timber products and wood based panels have indicated that besides intrinsic biological durability of the material the wetting and drying over time is also a key parameter. Attempts to quantify this role are leading to a range of data sets according to different methodologies. Besides solid timber of different wood species and plywood the correlation between laboratory test methods and actual time of wetness recorded in field experiments are also useful for wood treated with hydrophobers like organosilicon compounds.

Keywords: organosilicon compounds, hydrophobation, moisture dynamics, time of wetness, wetting ability

1. INTRODUCTION

The overall objective of this project is to extend the service life of constructions made of wood materials by providing water repellence to the materials thanks to "Si-based" solutions. Several studies explored already use of silane or siloxane as hydrophober for wood (De Vetter *et al.* 2010; Mai and Militz 2004; Ghosh *et al.* 2009 and 2012).

Wood is subject to water infiltration by both liquid and vapour. As the moisture content increases, the wood will swell until it reaches its maximum dimension at around its fiber-saturation point (FSP at about 30% moisture content). Dimensional changes not only result in volumetric differences but can also induce checking and cracking. Such cracks will then allow moisture to absorb easily and quickly into the wood. At some moisture content levels and depending on the time of wetness moisture will be present as free water, which in turn promotes the kick off and progress of wood decay.

Within the European framework of harmonisation of service life prediction tools it became obvious that durability based on the presence of active ingredients that function against biodegrading organisms is not the only critical parameter. Especially when focusing on end uses related to use class 3 and to some extent also to use class 2 the availability of water for decay fungi can be equally important. Hence, the material resistance of wood species, modified wood or preservative treated wood could be based not only on intrinsic or enhanced biological durability but could also be based on altered or improved moisture dynamics preventing the material of getting wet or staying wet. Time of wetness (ToW) is a critical parameter which is related to some moisture dynamic parameters. ToW can be determined with lab testing.

Material resistance is typically assessed by means of laboratory testing. Focussing on fungal decay as relevant for hazard class 3, it is clear that the biological agent requires a moisture level (over time) in combination with intrinsic nutritional quality or toxicity (referred to as durability). Current organism related testing (e.g. against Basidiomycetes) focusses on this intrinsic nutritional quality / toxicity level. However in a complementary way material resistance can also be based on how difficult a wood product gets and stays sufficiently wet. Socalled moisture related testing has been introduced recently and intends to differentiate wood materials based on moisture behaviour / dynamics. To some extent, these elements have been explored in detail within the European project PerformWood.

Additional to the material resistance parameter 'biological durability' there is a need for a classification based on moisture dynamics. Prior to establishing reference materials or products for this parameter it is clear that a relevant test procedure should be available. At UGent-Woodlab work on this topic was initiated earlier with focus on plywood. The objective was to link specific laboratory immersion tests with outdoor CMM data (Continuous Moisture Measurements - ToW concept). Since both wetting and drying parameters are important a floating test method was developed using first water permeability – absorption using contact with a water surface during 72h followed by water vapour permeability – desorption in a second step for 72h. To validate this test method the results were compared with field simulation in a CMM set up. Based on this success story with plywood a similar test protocol was developed for solid wood and has since been introduced to become a CEN standard method (prEN 16818, 2015). Further discussions related to testing without weekend impact and to include some longer periods for species with slow water uptake it was decided to prolong the test up to 144 hours absorption prior to desorption and to add a submersion test with open cross cuts. Meanwhile this method was part of a round robin as is reported at the IRG meeting in the paper by Brischke *et al.* (2014).

2. EXPERIMENTAL

2.1 Test methodology

2.1.1 Laboratory testing of moisture dynamics

To get insight in the moisture dynamics that have an impact on ToW one can consider both surface and end-grain phenomena to be relevant. Earlier developed methodology for plywood (Van den Bulcke *et al.* 2011) was based on a floating test to reveal how fast water enters through a wood surface and how easy it dries afterwards. In this concept reference was made to testing coatings according to EN 927 part 5 and the overall methodology was proposed earlier by Rapp *et al.* (2000). As solid timber is also prone to axial water uptake in particular when a timber construction allows for some water trapping a second method based on submersion was added. Both methods are briefly outlined in Table 1.

The basic idea was to start from stakes as provided for e.g. EN 252 in ground field testing having cross sections of 50 by 25 mm² and a growth ring angle close to 45°. The sampling procedure for the results presented in this paper focussed on having matched samples for both floating and submersion testing. A more detailed description can be found in prEN16818 (2015) and the related IRG paper by Van Acker *et al.* (2014).

Table 1: Floating and submersion tests to assess moisture dynamics

Parameter	Floating test	Submersion test
specimen cross section	50 by 25 mm²	
specimen length	50 mm	150 mm
edge sealing	yes	no
water penetration	one surface	whole specimens
absorption phase	1, 4, 8, 24, 48, 72, and 144 h	
desorption phase	1, 4, 8, 24, 48, 72, and 144 h	
preferred unit	g/m²	kg/m³

This methodology was also presented for a range of modified wood materials by Van Acker *et al.* (2015) at ECWM8.

2.1.2 Field testing of moisture dynamics

The continuous moisture measurement (CMM) set up as detailed in Fig. 1, was first installed to assess the moisture dynamics of plywood (Van den Bulcke *et al.* 2009). This methodology was already used to assess modified wood (Van Acker *et al.* 2015) and now it has been used to follow a set of organosilicon treated specimens. Specimens are identical to the ones used for the submersion test and edges were sealed. The specimens used for this simulated field test have the same dimensions as specimens intended for the submersion test as indicated in Table 1. After installation in May 2015 a specific rain event at the end of August 2015 was selected to analyse major differences.

Figure 1: The continuous moisture measurement (CMM) set up.

2.2 Materials

Scots pine sapwood specimens were vacuum impregnated using water based treating solutions containing 10 % active ingredient. All products under test have a commercial background from Dow Corning, not necessarily developed for wood hydrophobation. Results are presented for a selection of 3 products for which differences in moisture dynamics were observed.

The first product A is a non ionic emulsion containing a mix of silane, silicone resin and siloxane. The product B is a cationic emulsion of organo functional siloxane. The last product C is a siliconate, which is enables to test fully water soluble water repellent.
The three products enable to test different product concepts. Product A contains silane which are capable to react with silanol or hydroxyl groups. Product B structure provides high affinity for fibers while product C is a water soluble water repellent which reacts with carbon dioxide to cross-link and develop water repellency.

2.3 Calculation and expression of results

2.3.1 Laboratory testing of moisture dynamics

Although one can expect different absorption and desorption mechanisms resulting in short term (< 1 hour) and long term (> 6 days) results, the results presented here focus on the 144 h absorption and 144 h desorption. To allow some interpretation of the rate of water uptake and release the data collected were converted in some simple curves enabling to use the parameters of these for comparison. Van Acker and co-authors (2014) calculated mean values of 144 h absorption and desorption and ranked them both for the floating and submersion. Values after

144 hours of floating were considered high when over 5000 g/m² and corresponding values for desorption should then be over 2000 g/m².

The absorption curves fitted for both the floating test and the submersion test are based on the formula (Eqn. 1):

$$f(x) = ax^b \qquad (1)$$

When the b-value is close to 0.5 one can expect the a value to be a parameter related to the absorption coefficient. This is based on a linear relationship with the square root of time. Deviating values of b can point at special absorption phenomena like impact of capillary uptake. The a parameter is very good to assess the steepness of the linear area up to 4 or even 24 hours.

The desorption curves are anyhow somewhat more complex and were based on the formula (Eqn. 2):

$$f(x) = a + be^{\left(-x/c\right)} \qquad (2)$$

Van Acker *et al*. (2015) showed the potential to discriminate between species. Interpretation of the parameters here is also related to the 144 hour absorption values. The a value is the asymptotic value after drying and corresponds to the desorption value after 144 hours. The b value equals the amount of moisture that is released. Hence, the sum of the parameters a and b should be similar to the total absorption after 144 hours. The parameter c is corresponding with how fast the material dries, low c-values correspond with fast drying. High values of these 3 parameters correspond to high risk of accumulating water and hence an important time of wetness (ToW).

2.3.2 Field testing of moisture dynamics

Taking into account conditioned weight of specimens and calculation of starting moisture content based on moisture control references as well as recalculating mass recordings based on the impact of the edge sealant recordings, the specimens changing moisture content was recorded just before and after rain events by the end of August 2015 alongside the actual precipitation. A moderate rain event is considered to be between 2.5 and 7.6 mm/h. To avoid errors through shifting of readings by the load cell recalibration and reference standard mass recordings are performed on a regular basis allowing for confidence in the moisture content values.

3. RESULTS AND DISCUSSION

3.1 Laboratory testing of moisture dynamics

The reference blocks absorbed close to 10000 g/m² over the 144 hour period (Fig. 2). Of this water uptake all but some 500 g was gone during the 144 h desorption.

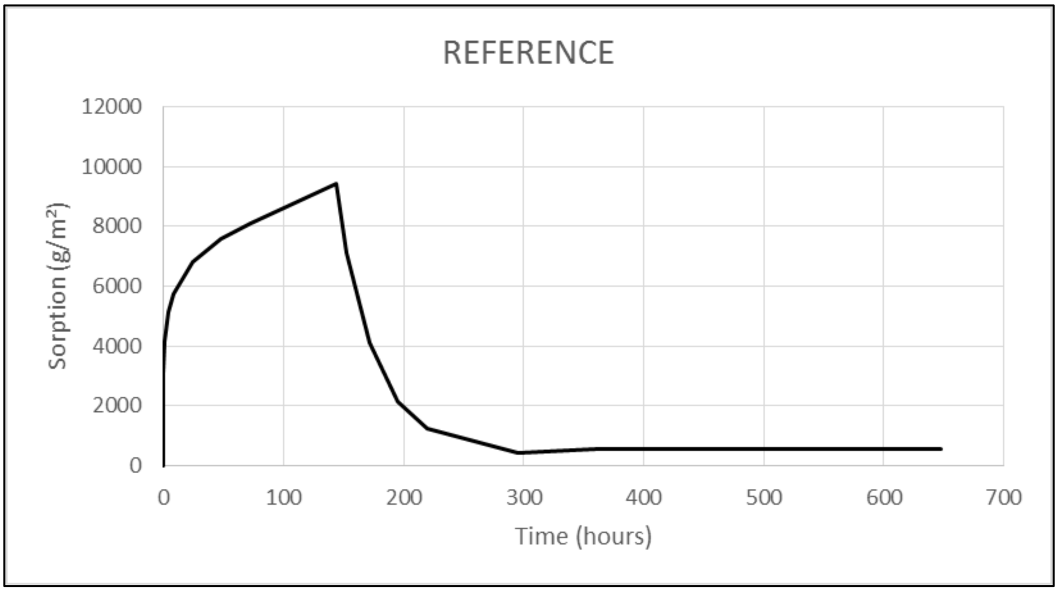

Figure 2: Absorption for 144 h and desorption for up to 3 weeks of floated Scots pine sapwood reference material.

In Table 2 details on relevant parameters are given for the 3 products presented. The a parameter is in line with the total absorption after 144 hours. The desorption values are in line with the absorption values indicating low risk for accumulating water leading to issues on ToW. The low c-value for product C however indicates fast drying.

Table 2: parameters derived from absorption and desorption of Scots pine sapwood treated with product A, B and C.

Product		**absorption**				**desorption**		
	Δ 144h	**f(x)=a*x^b^**		**est. 144h**		**f(x)=a+b*exp(-x/c)**		**est. 144h**
	(g/m²)	**a**	**b**	**(g/m²)**	**a**	**b**	**c**	**(g/m²)**
A	8630	1319	0.39	9320	568	8087	63	8655
B	6793	811	0.43	6741	364	6436	44	6800
C	10839	1815	0.38	11732	0	10995	32	10995

Fig. 3 is showing similar data for the 3 products as for the reference material in Fig. 2, however there is a clearly slower adsorption in the beginning for all treated material This delay is more prominent for product A than for product B and is less for product C. Overall on the total amount absorbed product A seems to alter the moisture dynamics of the Scots pine sapwood less than product B, while treated with product C an increased absorption is observed.

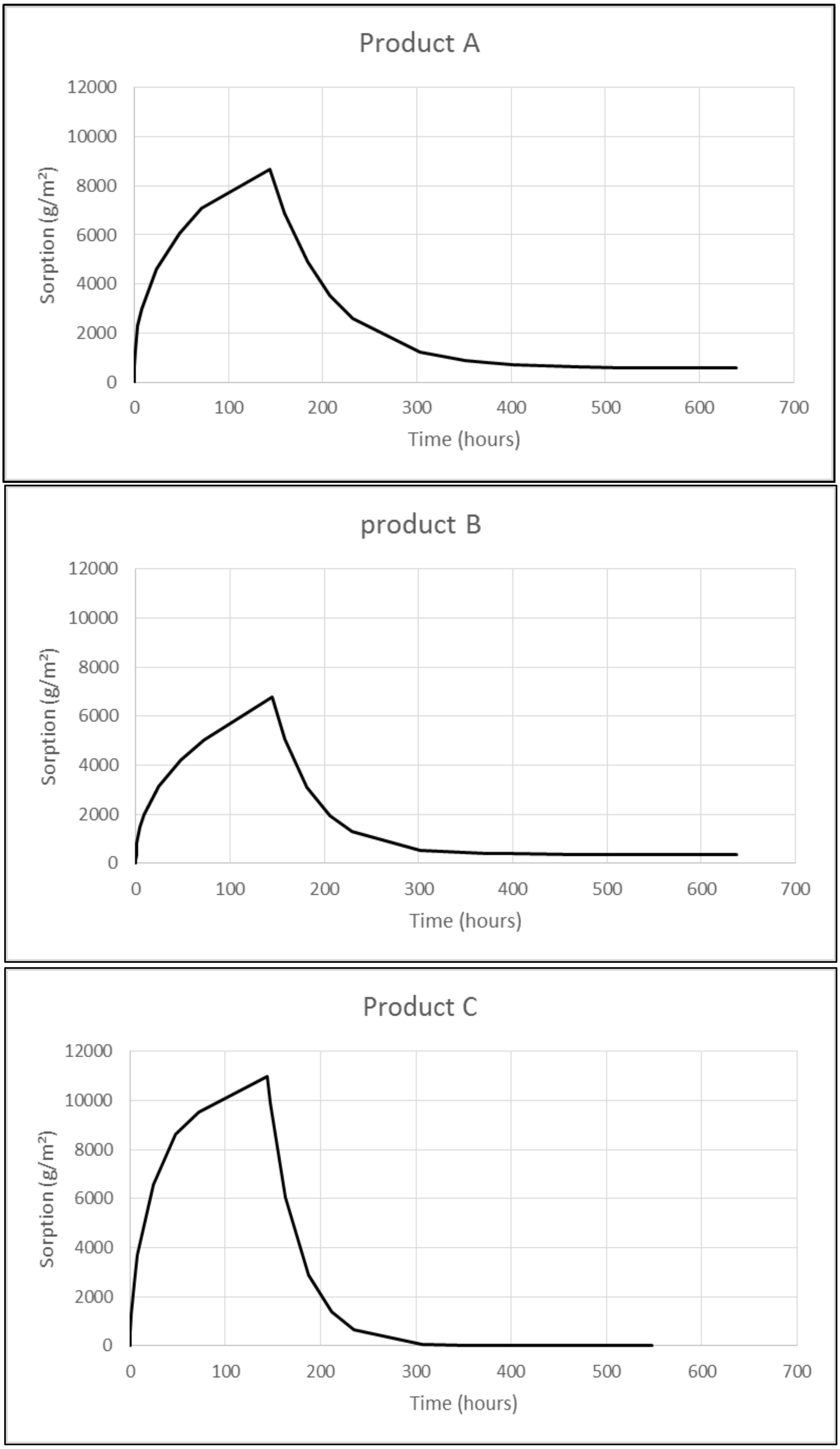

Figure 3: Absorption for 144 h and desorption for up to 3 weeks of floated Scots pine sapwood treated with products A, B and C.

3.2 Field testing of moisture dynamics

In Fig. 4 for each product 2 specimens are presented as well a reference to interrelate. Clearly all Scots pine sapwood treated with hydrophobing agents show lower moisture content than the reference. However the major differences between the performance of each of the products can be found in the reactivity to rain and the drying phase just after. Product C follows the same trend as the reference though at a lower moisture content. Product B treated wood does not differ from the reference for the short rain event on August 30th, but is not so sensitive to the longer rain event the next day. The product A induces lower absorption overall but also reveals somewhat slower drying. Comparing the result from the floating laboratory test (Fig. 3) with the results obtained here for this 2 day period and some intermittent rain events can be difficult and clearly some predictive potential related to ToW still needs to be checked.

4. CONCLUSIONS

The material resistance against decay can be based on the presence of active ingredients that slow down or prevent degradation by specific decay organisms, but in addition have also to be based on material properties that slow down wetting or limit staying wet. A methodology was developed to get as much information as possible on these moisture dynamics focussing on face water penetration valid for plank surfaces. The parameters derived are used to describe the moisture dynamics of a wood species and modified timber. A continuous moisture measurement during a rain event allowed to confirm earlier to this methodology and to discuss behaviour of modified wood (Van Acker et al., 2015).

It is possible to determine critical differences between products not even inducing a dramatic change in moisture dynamics. This methodology to assess wetting and drying characteristics should allow to optimize product performance in relation to time of wetness as one of the components to increase service life.

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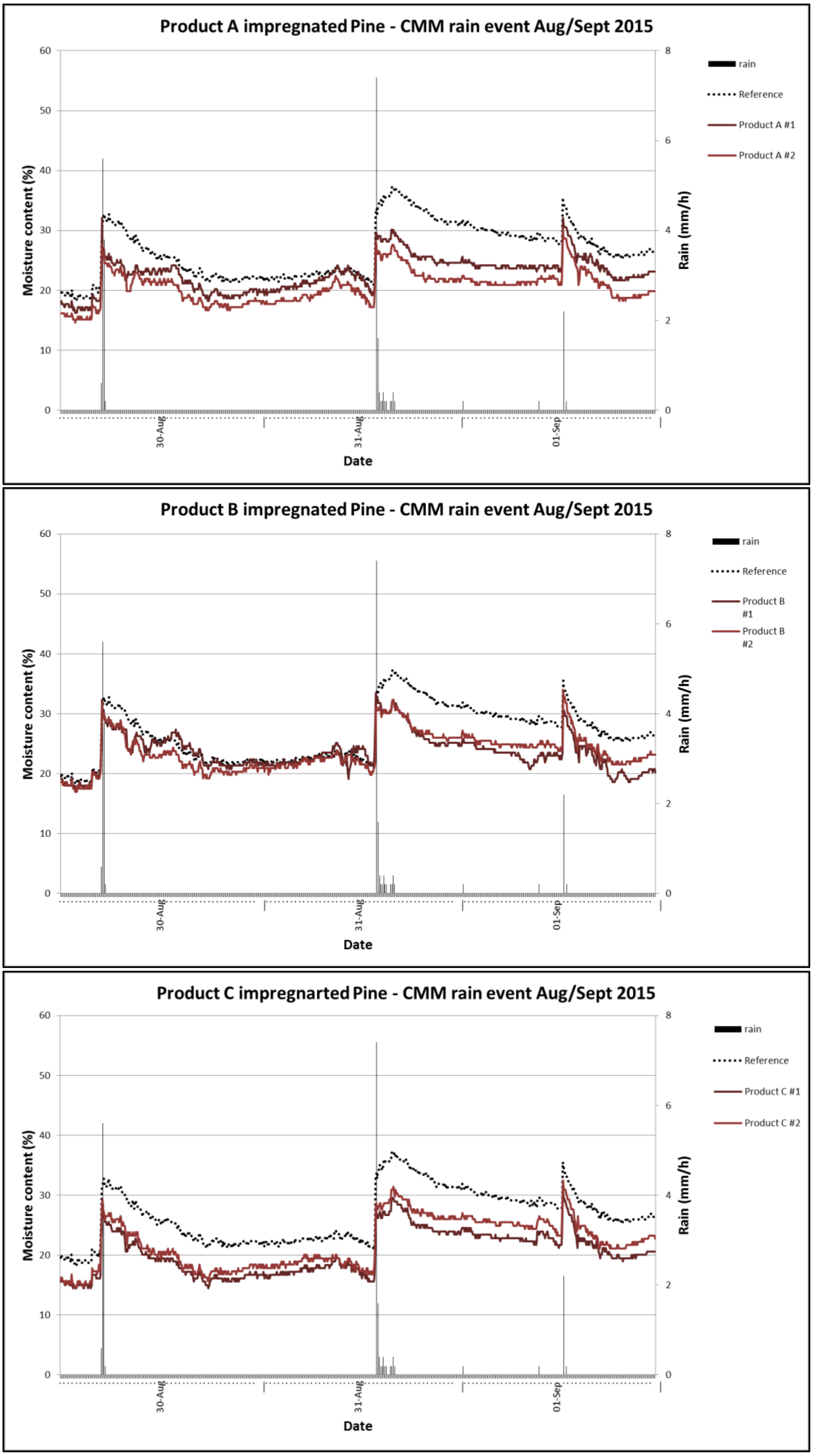

Figure 3: The continuous moisture measurement (CMM) of Scots pine sapwood treated with products A, B and C during rain events end of August 2015.

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