IMPROVING PERFORMANCE OF THERMAL MODIFIED WOOD AGAINST TERMITES WITH BICINE AND TRICINE

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Abstract
The desire to incorporate wood in modern construction has led to a considerable increase in the use of wood modification techniques, and especially thermal modification. However, thermally modified wood has poor performance against termites. The concept of using a combined chemical and thermal modification has been undertaken through the impregnation with either bicine or tricine prior to modification. This paper considers the effects of these chemicals on the activity of termites and considers their mode of action in terms of termite survival and on their effects on the symbiotic protists present within the termite gut.

Keywords:
Chemical modification, thermal modification, termites, buffers.

1 INTRODUCTION

Construction with timber is increasing in popularity, driven by sustainability agendas, ease of construction, aesthetics and human health. This increase in popularity coincides with European regulations, and particularly the European Green Deal, which was adopted by the European Parliament in January 2020. Within this policy, it was clearly stated that the policy "*encourages the promotion of timber construction and ecological building materials*". It then continues to state that the EU, through its policies "*believes that sustainably-sourced renewable materials will play an important role in the transition to a climate-neutral economy, and highlights the need to stimulate investments in the development of a sustainable bioeconomy where fossil-intensive materials are replaced with renewable and bio-based materials in, for example, buildings, textiles, chemical products, packaging, shipbuilding and, where sustainability can be assured, energy production*" and future activities should *"call for the efficient implementation of the EU Bioeconomy Strategy as part of the European Green Deal*". These new initiatives are seeing the use of wood in construction becoming more acceptable globally, resulting in their use in regions susceptible to decay from fungi due to high moisture levels, or from predating insects (Fig. 1).

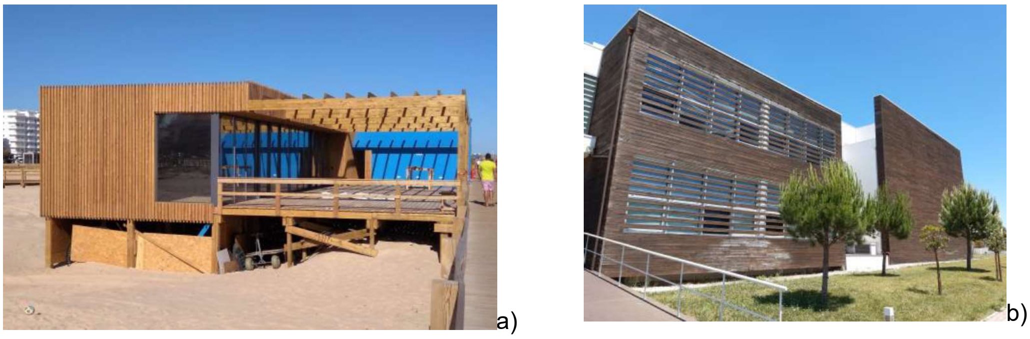

Fig. 1: Examples off thermal modified wood use a) in construction phase b) after circa 10 years of application (Photos: Lina Nunes).

Termites (Blattodea; formerly Isoptera) are consumers of cellulose and lignocellulose found in dead wood, grass, microepiphytes, leaf litter, and sometimes, cultivated fungi. Some 3,000 species of termites have been described, most having a tropical and temperate distribution across the USA, Central America, most of South America, southern Europe, Africa, Middles East, Southern Asia, Japan and Oceania, with the species most responsible for structural damage being *Coptotermes formosanus* Shiraki, *Coptotermes gestroi* (Wasmann) and *Cryptotermes brevis* (Walker) (Rust and Su, 2012). Of these 3,000 species, only 83 are considered to present a risk to wooden structures and furniture (Rust and Su, 2012). Europe lies on the border of traditional termite presence, but global warming may affect their distribution into more northern areas. This is shown in a detailed European distribution (Fig. 2, Kutnik et al., 2020), which shows the traditional range of a variety of *Reticulitermes* species, with cases having been reported in Paris, Hamburg and in Devon, England.

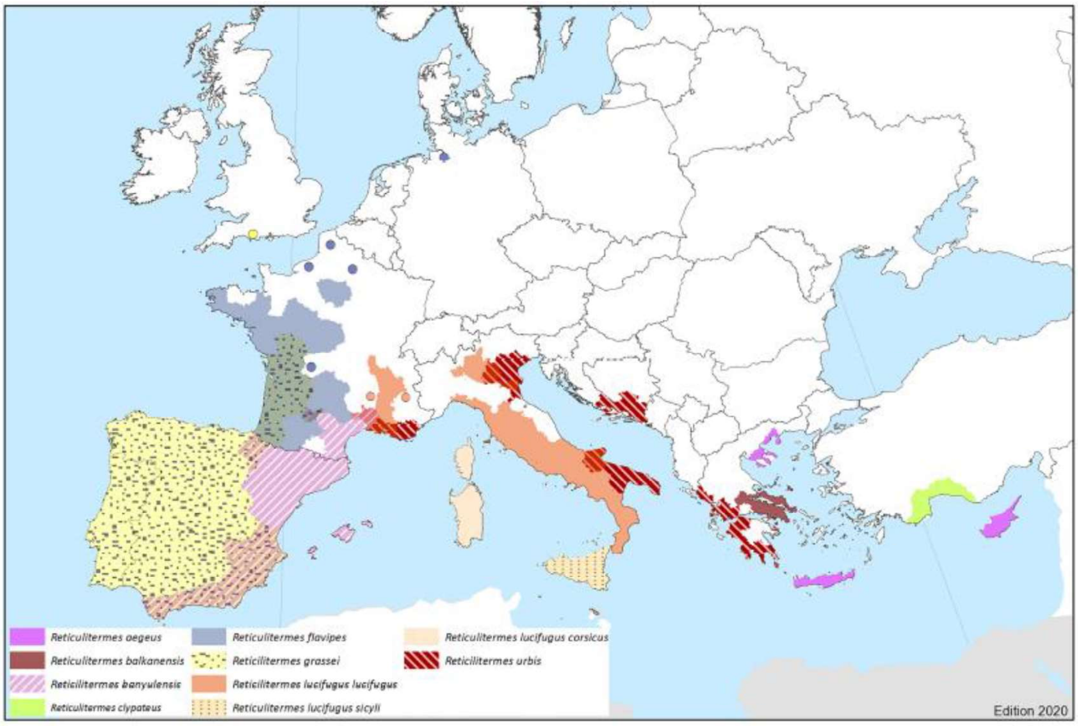

Fig. 2: European distribution of termite species (courtesy of Magdalena Kutnik).

The common method for protecting wood products against termite attack has been through chemical treatment, and whilst this remains the favoured method, the introduction of wood modification has been an alternative to conventional wood preservation in many countries. In particular, thermal modification has been adopted as an exterior finishing by many architects and specifiers (Fig. 1). However, it is known that thermally modified wood may exhibit different degrees of resistance to termites, according to the wood product and heat treatment technique, among other factors (Unsal et al., 2009; Oliver-Villanueva et al., 2013; Brito et al., 2020), but is generally recognised as low (Salman et al., 2017).

2 COMBINED CHEMICAL AND THERMAL TREATMENT OF WOOD

In order to improve the properties of thermally modified wood, there have been attempts to integrate chemical modification methods. Examples include the use of wax impregnation (Humar et al., 2017; Zhang et al., 2020) and resins (Sun et al., 2013; Behr et al., 2017). The potential of combining chemical modification with thermal modification was demonstrated by the work of Mubarok et al. (2019), with the impregnation of compounds such as vinylic-polyglycerol, vinylic-glycerol, and maleic anhydride followed by thermal modification as a means of achieving a non-biocidal anti-fungal and anti-termite wood treatment.

The darkening of wood during thermal modification is well-known and is thought to be the result of a Maillard reaction, which is typically a reaction between an amine and a reducing sugar. This mechanism was proposed by Hauptmann et al. (2015) for the reaction of xylose with tricine. By better understanding the depolymerisation/degradation process within the hemicellulose, it may be possible to apply Maillard-type reactions and react the sugar moieties within the cell wall matrix, instead of being volatilized. This was studied in more detail by Peeters et al. (2018).

The potential of tricine as a potential treatment was explored further (Jones et al., 2019), whereby wood samples were treated with tricine and bicine respectively before thermal modification at a slightly reduced temperature (160 °C), due to the potential of thermal degradation of the chemicals at temperatures above 180 °C. This combined modification resulted in a slight limitation of mechanical property losses, whilst evaluation of colour data seemed to suggest there was an effect from the inclusion of the amine compounds resulting in a Maillard-type reaction. Additional studies by Fourier transform Infra-red (FTIR) spectroscopy (Popescu et al., 2020) confirmed the reaction had occurred to some extent. This current study, carried out as an extension of these previous works, aimed to ascertain any effect against termite attacks, given that the thermal modification of wood alone offered little or no protection against termite attack (Salman et al., 2017). Both tricine and bicine are known as zwitterionic buffers, and more specifically Good's buffers (Good et al., 1966; Ferreira et al., 2015).

3 EXPERIMENTAL METHODS

3.1 Treatment of Wood

Wood samples of spruce (*Picea abies* (L.) H. Karst) and beech (*Fagus sylvatica* L.) of dimensions 30 x 10 x 10 mm (LxRxT, Gorenjska, Slovenia) were treated with solutions of tricine and bicine (obtained from Fisher Scientific, Hampton, NH, USA), using a vacuum impregnation method: the samples were placed in containers and weighed down, 1 M solutions of bicine or tricine in distilled water were respectively added; the impregnation schedule followed the full cell process, 20 min of vacuum of 10 kPa followed by two hours of overpressure at 900 kPa and at the end for 10 min of vacuum of 10 kPa. Before and after the impregnation, the samples were dried and weighed to determine uptake. The treated samples were allowed to dry under low heating conditions (50 °C).

Once dry conditions had been achieved, samples were then thermally treated using a modified process to the one described by Rep et al. (2012), whereby a reduced maximum temperature of 160 °C was used (instead of the conventional 180–230 °C employed for thermal modification of wood) under vacuum. This was to minimise the thermal degradation of the bicine and tricine respectively at temperatures around 180 °C. Spruce and beech samples were also subject to the same heat treatment process of 160 °C without any impregnation.

The following combinations of treatments were evaluated against subterranean termites for both wood species: (1) untreated; (2) heat-treated (HT); (3) bicine; (4) bicine and heat treatment (bicine HT); (5) tricine; (6) tricine and heat treatment (tricine HT).

3.2 Effects on Subterranean Termites

Subterranean termites belonging to the species *Reticulitermes grassei* Clément, and to three different colonies, were captured in a pine forest (*Pinus pinaster* Aiton), in Sesimbra, Setúbal district of Portugal. Each colony was captured on a dead wood log (approximately 1 m long and 0.08 m diameter) more than 100 m apart. Logs were brought to the laboratory and kept in a conditioned room at 24 °C ± 1 °C and 80% ± 5% relative humidity. Groups of 150 termite workers were captured manually and established in 200 mL glass jars with moistened sand (Fontainebleau sand and water; 4:1 v/v) as substrate. The previously treated spruce and beech wood was offered to the termites as food, three replicates per treatment. An example of the experimental set-up is shown in Fig. 3.The test was run for four weeks in the described conditions. Maritime pine test specimens with the same dimensions were also included as internal virulence controls (EN117, 2012; Esteves et al., 2017).

Fig. 3: Example of experimental set-up, based on a modified EN 117 test.

The initial moisture content of the blocks was measured in sets of three additional replicates per treatment and these values were used to determine the theoretical initial dry mass (*IDM*) of the exposed specimens. At the end of the trial, the final moisture content was recorded, and the mass loss was obtained according to Equation (1).

$$\% \text{ mass loss} = (FDM - IDM)/IDM \times 100 \qquad (1)$$

Where: *FDM* is the dry mass of the block at the end of the test.

The survival rate (%) of the termites was also recorded. All wood blocks were graded according to termite attack using the scale: 0 = no damage; 1 = attempted attack; 2 = slight damage; 3 = superficial and inner damage; 4 = heavy inner damage (EN117, 2012).

4 RESULTS AND DISCUSSION

The average results for survival of termites, along with the mass loss and grade of attack of samples are presented in Tab. 1. The survival of the termites and the mass loss (Fig. 4) showed some differences between the treatments although a high variability of the results was observed for some treatments Differences might be partially explained by the species tested as the impact of bicine and tricine seem to be higher on spruce (Duarte et. al. 2021).

Tab. 1: Average results of efficacy of treatments against R. grassei termites. Standard deviation in brackets.

Treatment	Wood	Termite Survival (%)	Mass Loss (%)	Grade of Attack
Untreated	Spruce	88.4 (6.3)	11.3 (0.8)	4.0 (0.0)
	Beech	82.0 (9.7)	7.7 (0.9)	4.0 (0.0)
	Maritime Pine	84.4 (7.4)	8.0 (3.3)	4.0 (0.0)
HT	Spruce	90.8 (4.5)	11.8 (2.1)	4.0 (0.0)
	Beech	87.2 (2.7)	8.2 (0.8)	4.0 (0.0)
Bicine	Spruce	64.2 (38.9)	2.6 (1.1)	3.0 (0.0)
	Beech	38.0 (22.0)	3.7 (0.3)	3.0 (0.0)
Bicine HT	Spruce	21.1 (18.6)	2.9 (1.6)	2.7 (1.5)
	Beech	57.6 (47.0)	4.4 (1.4)	3.0 (0.0)
Tricine	Spruce	11.3 (17.4)	2.4 (1.2)	3.0 (0.0)
	Beech	71.4 (17.2)	5.8 (0.3)	3.7 (0.6)
Tricine HT	Spruce	62.3 (31.2)	10.1 (1.9)	4.0 (0.0)
	Beech	79.9 (16.8)	6.2 (0.0)	4.0 (0.0)

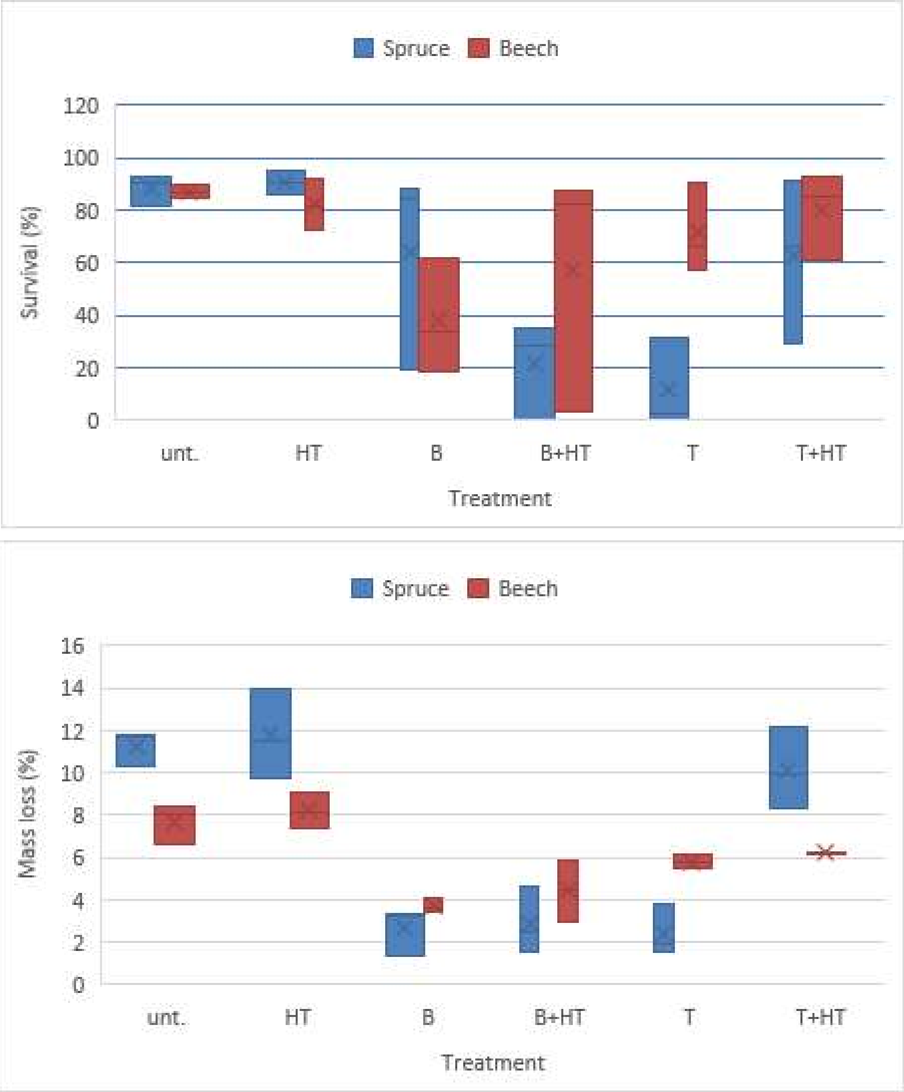

Fig. 4: Results of survival and mass loss highlighting the variability of the results and the differences between species tested.

As indicated earlier (Salman et al., 2017), the durability of thermal modified wood is recognised as low and the results of the present work are in accordance with that perception. However, bicine and tricine treatments alone had a clear influence on the survival of the termites. For spruce, it was observed a lower survival rate for tricine + oven dry and bicine + oven dry + HT, together with a lower mass loss and attack grade, which may indicate a detrimental effect of those treatments (and also bicine + oven dry) on termite fitness. For beech, bicine treated wood (both bicine + oven dry and bicine + oven dry + HT) showed a clear negative effect on termite survival and wood mass loss and attack grade. As yet unpublished results suggest that the combination of bicine and heat treatment resulted in a combined weight increase of treated samples greater than would be expected from the individual treatments, with the weight increase actually being greater than that noted for treatment with bicine alone. This provides further indication of some Maillard-type reaction occurring, whereby components of wood normally lost during thermal modification have been retained via interaction with the impregnated bicine.

The effects on termite survival are not thought to be as a result of any termiticidal effect, given that tricine and bicine are known agents as non-fermentable buffers, with tricine used in animal tissue cultures and bicine in the manufacture of primary cells (Soni and Kapoor, 1981; Armenante et al., 1993). Both compounds are known to have a slightly basic nature, with pH values of 7.4–8.8 and 7.6–9.0 for tricine and bicine, respectively. It is well-known that termites have a range of symbiotic fauna within their digestive gut, and the evaluation of these treatments on these symbiotic protists has been recently published (Duarte et al., 2021). Therefore, these studies offer two potential modes of action:

- The compounds chemically inhibit the activity of selected protists in the termite gut; and/or
- The compounds alter the acidity of the gut environment.

The latter suggestion is possible given the pH of both chemicals is slightly basic in nature (bicine: 7.6–9.0; tricine: 7.4–8.8), which may influence the hindgut pH of lower termites, known to be 6–6.5, for *Reticulitermes lucifugus* (Rossi) paunch (Bignell and Anderson, 1980), and thus estimated to be similar for R*eticulitermes grassei* Clément. As the termite hindgut is a highly structured environment, comprising micro-niches with graduated acidity and oxygen/hydrogen levels, any alteration on the pH may lead to the disruption of the hindgut chemical stability (Brune and Friedrich, 2000), resulting in changes in the termite symbiotic community.

In the recent study (Duarte et al., 2021), the nine identified hindgut protists in *R. grassei* were studied, with significant decreases in the abundances of most protists being observed, particularly when compared to the termites fed on maritime pine reference samples, except for the protist *Holomastigotes elongatum* Grassi, a protist belonging to Hypotrichomonadidae family and *Pyrsonympha* morphotype 2. Bicine HT spruce-fed termites showed a significant decrease in *Trichonympha* sp., *Pyrsonympha* morphotype 1, n3 (*Dinenympha gracilis*, Leidy and H. elongatum. The tricine-treated spruce-fed termites showed significantly lower abundances of the protists: *D. gracilis* and *Microjenia hexamitoides*, Grassi compared to the untreated spruce-fed termites.

4 SUMMARY

Bicine and tricine treatments alone had an influence on the survival of the termites, whilst the combination of bicine with a mild thermal modification had a significant impact of termite survival. Similar results were not found with the combination of tricine treatment and thermal modification. This seems to suggest that the chemical changes previously identified by infrared spectroscopy and chemometric methods (Popescu et al., 2020) suggesting reactions between the functional groups in the wood and side chains from tricine (via a chain displacement mechanism) did not impact termite survival.

The characterisation of the chemical behaviour of both of these substances inside wood and after heat treatment will allow the understanding of the mode of action towards the symbiotic fauna of the subterranean termites, as the results obtained in this study seem promising. Thus, further investigations into these mechanisms are warranted. The evaluation of bicine and tricine as possible treatments against fungal decay is also under current consideration.

5 ACKNOWLEDGMENTS

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SORPTION IN BIO-BASED BUILDING MATERIALS: IMPROVEMENT OF THE LOCAL KINETICS MODEL AND APPLICATION TO THE SIMULATION OF THE ISOBIO WALL STUDIED IN A DEMONSTRATOR

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Abstract

A bio-based multilayered wall has been developed in the framework of the European ISOBIO project. A key point was to be able to perform proper simulations of the hygrothermal transfers occurring through the wall: local predictions are of first importance to characterize the behavior of the wall and thereafter its ability to ensure comfortable hygrothermal conditions inside buildings. Previous studies proved that the conventional assumption of an instantaneous equilibrium between local relative humidity and water content according to the sorption isotherm is not relevant for bio-based porous materials, where in practice a slow sorption kinetics occurs. In the present study, an improved expression of the local kinetics is proposed and validated by sorption experiments. Then, the case of the reference ISOBIO wall submitted to a given real climate (Wroughton, HIVE demonstrator, UK, Feb 2018) is investigated: simulations based on the classical approach (TMC code) or considering the sorption kinetics (TMCKIN code) are compared to the measurements. It appears that TMC simulation underpredicts the relative humidity dynamics whereas TMCKIN simulation considering the improved expression of the local kinetics is in good accordance with measurements. Finally, an alternative configuration of the ISOBIO wall is numerically studied, showing that a better hygric behavior can be obtained.

Keywords:
Bio-based materials; Local kinetics; Sorption; Hygrothermal transfer; Demonstrator; Modeling

1 INTRODUCTION

In the framework of the European ISOBIO project (featuring the slogan "naturally high-performance insulation"), a multilayered wall made of bio-based materials involving very low carbon footprints and high insulating properties has been developed. One of the key points of this project was to be able to perform reliable simulations of the hygrothermal transfers occurring inside this ISOBIO wall. The main difficulty with such simulations is to correctly describe hygric phenomena in bio-based materials, as shown in (Reuge et al., 2020a; Reuge et al., 2020b).

Water sorption in porous media involves various phenomena: the transport is ruled by vapor / liquid water diffusion and the water sorption by the equilibrium isotherms of adsorption / desorption coupled with hysteretic phenomena. Thus, conventional approaches describing hygrothermal transfers in building materials are based on the assumption that for a given local relative humidity φ, the corresponding equilibrium local water content w is reached instantaneously (Künzel, 1995). However, the aforementioned models are not sufficient to provide a good description of the hygric state in these porous media. This was shown for bio-based materials: in (Nyman et al., 2006) for cellulosic materials, in (Frandsen et al., 2007) and (Eitelberger and Svensson, 2012) for wood, in (Alexandersson, 2016) for paperboard, by Reuge et al. in (Reuge et al., 2020a; Reuge et al., 2020b) for ISOBIO materials and in (Reuge et al., 2019) for various hemp-lime concretes, but also for more classic compounds such as cements (Johannesson and Nyman, 2010; Jansen et al., 2016). Several expressions for a local kinetics of sorption can be found in the aforementioned references. A kinetic is usually expressed as a kinetic constant multiplied by a driving force. In most of the aforementioned studies, the authors have proposed to express the driving force as a function of local relative humidities with more or less success. The problem is that they consider an equilibrium relative humidity which is a priori not known for a local approach and / or they have to change the value of the kinetics constant according to the operating conditions. Actually, it seems more natural to express the driving force as a function of w, as done in (Nyman et al., 2006; Johannesson and Nyman, 2010).

In our aforementioned previous studies, various bio-based materials were studied and it was demonstrated that the classical approach led to patent inconsistencies such as a water vapor / liquid water equilibrium reached too fast at the local scale and at the sample scale and / or underestimation of the local φ dynamics. Thus, it has been established that a local kinetics of sorption exists (from water vapor to liquid water and inversely) which can be slow compared to the diffusive phenomena. In this study, an improved expression of the local kinetics of sorption is proposed and validated by experimental results allowing to obtain better results from the simulations for small time scales (from seconds to few hours).

Moreover, this is the first modeling work studying a fully bio-based multilayered wall subjected to a real climate. Various studies have already been carried out considering only one bio-based layer. In (Moujalled et al., 2018), the effect of real climatic conditions (Périgueux, FR) is studied on a building with a wall of three layers: a hemp-lime concrete but with classic internal and external renders. In (Piot et al., 2015), the authors have also studied a three-layered wall (Savoie, FR) in real climatic conditions: a hemp concrete wall but also with classic internal and external renders. In the studies (Lelievre et al.,2014) and (Colinart et al., 2016; Rahim et al., 2016; Fabbri and McGregor, 2017), the authors have studied the response of hemp concrete panels to controlled indoor and outdoor conditions. In most of these studies, simulations led to patent underestimations of the φ dynamics.

The first part of this study is a summary of the classic hygric and thermal properties measured on samples of ISOBIO wall materials. Some classic models are used and adjusted to describe these properties evolutions as a function of water content (*W*) or relative humidity (*RH*). Then, governing equations describing the hygrothermal transfers are given. In the following part, the expression of the sorption rate previously defined is recalled and a fine modeling of the global kinetics of sorption measurements is used to define an improved expression. Then, a hygrothermal study is performed at the wall scale in a demonstrator and an alternative configuration of the ISOBIO wall is finally proposed.

2 ISOBIO MATERIALS AND HYGROTHERMAL PROPERTIES

2.1 ISOBIO wall configuration

The reference ISOBIO wall is made of the layering of several materials / panels: a BCB^{TM} lime-hemp render (BCB), a CAVAC^{TM} Rigid insulation panel made of hemp shiv and an organic binder (CAV), a Biofib Trio flexible insulation panel from CAVAC^{TM} made of hemp flax and cotton (BIO1), an OSB3 panel, a ProclimaTM INTELLO membrane (INT), a second Biofib Trio flexible insulation panel (BIO2), a Lignicell CSB^{TM} panel made of compressed straw (CSB) and a CLAYTEC^{TM} clay-hemp plaster (CLA). Note although a timber frame supports this wall, this will be ignored in the simulations. This ISOBIO wall and layers thicknesses are shown in Fig. 1.

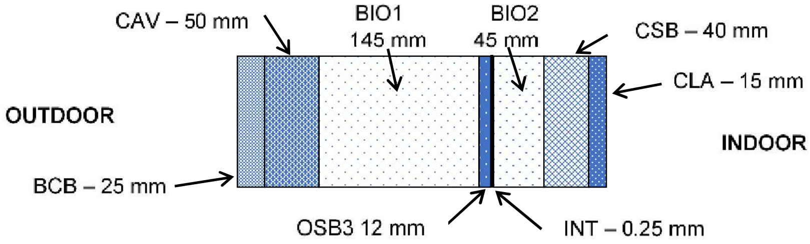

Fig. 1: ISOBIO wall configuration

2.2 Hygrothermal properties

This section summarizes the values of the properties needed to perform the simulations, *i.e.*, the materials densities ρ_0, porosities ε_0, vapor diffusion resistance factors μ_0, thermal conductivities λ_0 and specific heat capacities Cp_0: they are reported in Tab. 1. While most of them come from (Collet et al., 2019) where experimental methods and measurement are provided, a few others come from trustable technical sheets.

The isotherms of adsorption of representative samples of ISOBIO materials (CAV, BIO, OSB3 and CSB) have been determined in (Collet et al., 2019). The moisture storage functions of the other ISOBIO materials come from technical sheets and / or Fraunhofer Institute for Building Physics (F-IBP) databases (BCB, INT and CLA).

2.3 Models of properties

The appropriate models of properties needed for the simulations have been studied in (Reuge et al., 2020a). For the isotherms of adsorption, the Van Genuchten model (VG) (Van Genuchten, 1980) has been used. It is claimed valid even near the liquid water saturation state and is expressed as follow:

$$W_{eq}(RH) = W_{sat}\left[1+\left(-h\ln(RH)\right)^{\eta}\right]^{1-1/\eta} \tag{1}$$

where W_{eq} is the sample water content at equilibrium, W_{sat} is the maximum water content in the sample, *h* and η are adjustment coefficients. The values of these adjustment coefficients are given in Tab. 1. The hygric dependence of the vapor diffusion resistance factors μ has been neglected except for the Proclima INTELLO because this is a hygrovariable membrane evolving very significantly as a function of *RH*. The data provided by Proclima / F-IBP have been fitted by a logistic power law (Reuge et al., 2020a): $\mu = 1/\left(a+b.RH^{c}\right)$, with: $a = 7.3310^{-6}$, $b = 1.8.10^{-3}$ and $c = 7.644$. Finally, the hygric dependence of the thermal conductivities has been described by the self-consistent scheme (Collet and Pretot, 2014), which takes the following expression:

$$\lambda(W) = \lambda_s\left\{1+\varepsilon_0\left[\frac{1-\varepsilon_0}{3}+\left(3+\left(\frac{W}{1000\varepsilon_0}\right)\left(\frac{\lambda_a}{\lambda_w}-1\right)\right)\cdot\left(3\left(\frac{\lambda_a}{\lambda_s}-1\right)-\frac{W}{1000\varepsilon_0}\left(\frac{\lambda_a}{\lambda_w}-1\right)\frac{\lambda_w}{\lambda_s+1}\right)^{-1}\right]^{-1}\right\} \tag{2}$$

where λ_a (0.025 W.m^{-1}.K^{-1}) is the air thermal conductivity and λ_w (0.6 W.m^{-1}.K^{-1}) is the liquid water thermal conductivity. The coefficient λ_s has been adjusted such as $\lambda(W=0)$ equals λ_0: its values are given in Tab. 1.