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The metrological approach: a major key factor for the accreditation and continuous improvement of the wood preservation laboratory of Cirad

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Abstract. Since 2006, the wood preservation laboratory of Cirad is accredited by COFRAC (French accreditation committee – accreditation No. 1-1686) for tests on the durability of wood and wood-based products and on protective efficacy of wood preservatives and termite control products. The metrological approach adopted by the wood preservation laboratory is a key factor on the continuous improvement of practices. Tests to determine the resistance against wood-destroying biological agents are the most difficult of all wood analysis tests. They are aimed at assessing and quantifying the impact of living organism, such as fungi and termites, on a lignocellulosic material. The extent of variability of this impact, which in turn is linked with the diversity of these organisms and of the material, can be readily determined. The validity and reliability of the findings therefore depend directly on the quality of the metrological process, including the choice of measurement devices, their management and compliance with international standards.

Keywords: Metrology; accreditation; traceability; uncertainty; biological tests

1 Introduction

The Cirad wood preservation laboratory is accredited by COFRAC (accreditation No. 1-1686) according to ISO 17025 [1] standard for tests on durability of wood and wood-based products and on the protective efficacy of wood preservatives according to the European standards: EN113 [2], XP ENV 12038 [3] (fungal tests), EN 117 [4], EN 118 [5] (termite tests) and EN 73 [6], EN 84 [7] (associated accelerated ageing procedures). It is also accredited to test termite control products according to the French standard XP X 41-540 [8] and its associated ageing procedure XP X 41-542 [9].

The approach adopted by the wood preservation laboratory is reflected by the type of tests that is carried out as part of research projects or at the request of private operators. Tests to determine the resistance against wood-destroying biological agents are the most difficult to perform amongst wood qualification tests, such as mechanical or physical tests. They are aimed at assessing and quantifying the impact of wood-destroying organisms on lignocellulosic material, whether it is treated or not. The extent of variability of this impact, which in turn is linked with the diversity of these organisms and of the material heterogeneity, can be readily determined.

In order to evaluate, as precisely as possible this impact (i.e. biological degradation), the laboratory possesses an equipment pool of 60 measurement devices, including 14 measurement standards allowing the traceability to international standards. This pool is managed using the SPLIT4® software. The physical quantities involved are: length, angle, weight, temperature, hygrometry, pressure, volume, air flow.

To give an overview of the singularities of our field of research, the case study of the EN 117 [4] termite test will be described from the 1st step (wood selection) to the test results, and the major metrological particularities will be underlined.

This European standard specifies a method for the determination of the toxic values of a wood preservative against *Reticulitermes* species (subterranean European termites). It allows the determination of the concentration at which the product completely prevents attack by these termites of impregnated wood of a susceptible timber species.

In short, Scot Pine sapwood (non durable timber) is treated with a wood protection product using different concentrations. The treatment is done through a vacuum-pressure device and the quantity of product introduced into the wood (retention) is measured. After drying, and eventually accelerated ageing (EN 73 [6] evaporation or EN 84 [7] leaching), the treated wood blocks are exposed in culture flask to the attack of 250 termite workers. After 8 weeks exposure in controlled conditions ((27 ± 1) °C, >70%RH), the wood samples are removed from the flasks and (1) the survival of the workers is determined, (2) a visual rating of the wood samples is carried out to evaluate the degree of attack.

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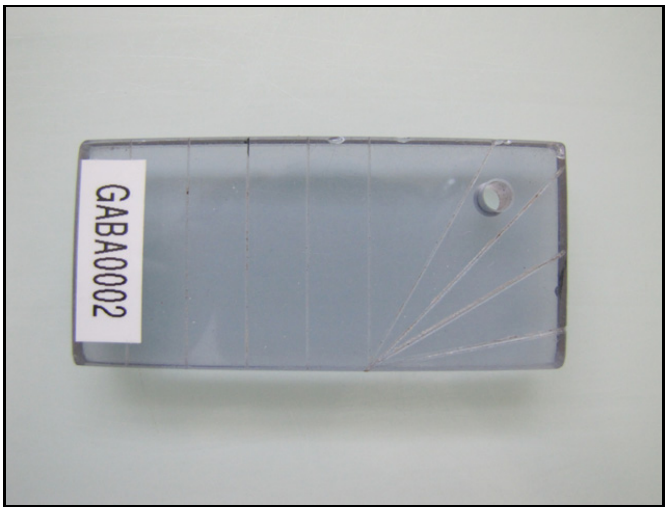

Fig. 1. Test gauge for wood sample selection (±1 mm, ±1°).

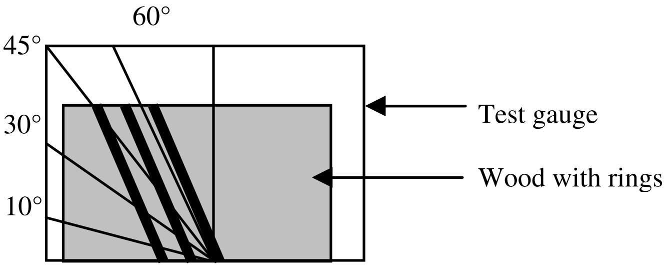

Fig. 2. Checking the orientation of rings according to EN 117 [4] standard.

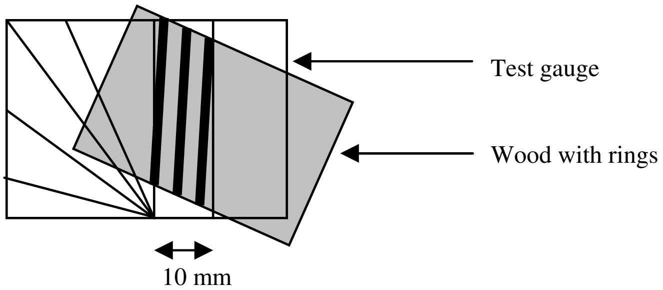

Fig. 3. Checking the number of rings for 10 mm according to EN 117 [4] standard.

In this paper, the crucial steps of this test will be described, as well as the metrological associated issues: wood selection, counting the number of termites, visual quotation of the wood samples.

2 Experimental arrangements

The different devices and experimental set up are shown in Figures 1–8.

3 Wood selection

This first step is very important. The standard imposes the timber to use (Scot Pine sapwood) of a given quality

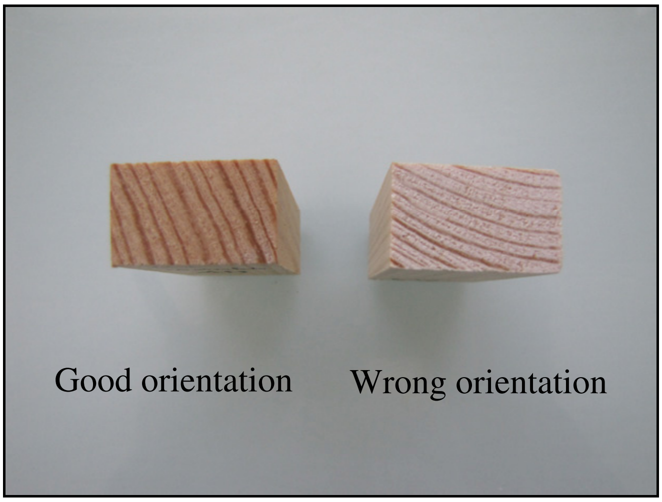

Fig. 4. Examples of orientation of annual ring in sapwood samples.

Fig. 5. Termite worker.

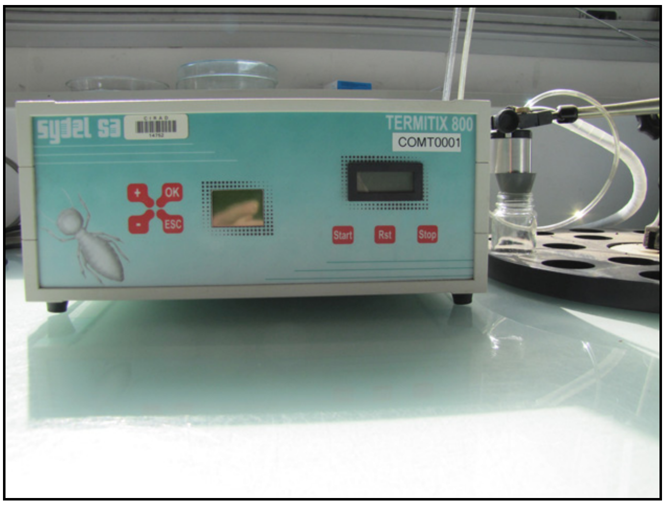

Fig. 6. Apparatus to count the termites, further so called "termite-meter".

Fig. 7. Culture flasks (test device) with termites.

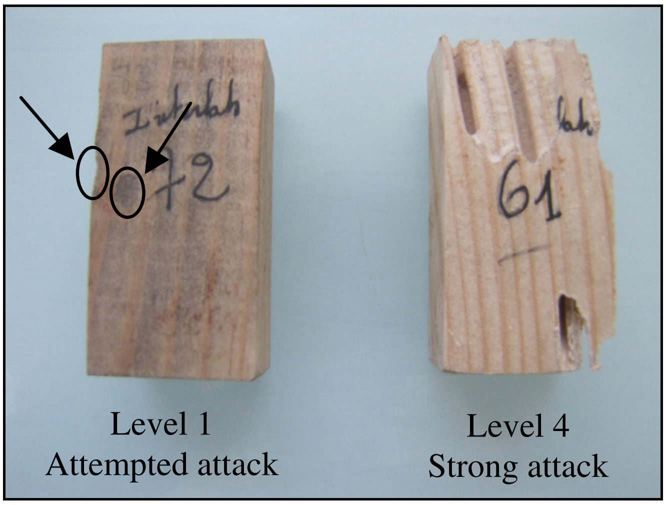

Fig. 8. Examples of visual rating.

(no cracks, resin, previous biological attack, treatment nor drying above 60 °C, ...), the dimensions, the ring orientation, as well as the proportion of latewood (less than 30%). These selective criteria aim to lower as much as possible the variability inherent to the biological matrix.

Wood is a hygroscopic material which shall be conditioned at a given atmosphere prior to any measurement (length or weight). Here, the dimensions of each test specimen after reaching equilibrium in a conditioning chamber ((20 ± 2) °C, (65 ± 5)%RH) measured with a fitted caliper shall be:

- length (0.0 ± 0.5) mm in the longitudinal plan of the wood;
- width (25.0 ± 0.5) mm in the radial plan;
- thickness (15.0 ± 0.5) mm in the tangential plan.

Furthermore, the wood shall be exclusively sapwood containing little resin and having between 2.5 annual rings per 10 mm and eight annual rings per 10 mm. The annual rings shall have a contact angle of greater than 10° to the broad faces (radial plan) of the test specimens. To verify proportion and orientation of annual rings, we have developed a test gauge (Figs. 1–4) that facilitates the work of operators both when the wood is cut (from the tree log to the plank, and then to the samples) and selected for the test.

Once this first selection is over, wood must be weight (with a fitted balance) to determine the density. Initial weight and density of the samples are important for the product retention calculation.

4 Termites

Once the wood is treated, dried and stabilized at (20 ± 2) °C, (65 ± 5)%RH, it has to be exposed to the attack of 250 termite workers (Figs. 5 and 7). For a complete evaluation of a product destined to become a certified wood preservative product, 40 devices representing 10 000 termites, are necessary.

To avoid the tedious job of counting the termite by hand, we have developed a device for counting termites (Fig. 6). This termite-meter is equipped of a suction pump to aspire the termite that go through a flexible pipe and in front of a photo-sensible cell that will count them. The 250 termites are stocked in a small beaker before being introduced in the test flask.

This prototype has been qualified for different parameters:

- the aspiration speed was chosen carefully because it must not affect the survival rate or vitality of the termites;
- the detection of the photo-sensible cell. First, it was checked that the numbered given by this cell was exact. Secondly, it was verified that only the termites were detected, and that the little particles (sand, soil) were ignored.

This reliable apparatus generates an average time saving of 50% for a test set up.

5 Visual rating

After 8 weeks of biological exposure, the wood samples are removed and the termites remained alive are counted (by hand for this step). To be declared valid, the virulence controls (untreated pine sapwood exposed in the same manner as described previously) must present a termite survival of 50% and a strong level of attack (rating 4).

Any evidence of attack is classified by its locality, its extent and its depth. The results of this examination (Fig. 8) are expressed in accordance with the following schedule:

- level 0: No attack;
- level 1: Attempted attack;
- level 2: Slight attack;
- level 3: Average attack;
- level 4: Strong attack.

Each level of attack can be a combination of 2 criteria. As an example, level 1 is reached for:

(i) superficial erosion of insufficient depth to be measured on an unlimited area of the test specimen; or
(ii) attack to a depth of 0,5 mm provided that this is restricted to an area or areas not more than 30 mm^2 in total;
(iii) a combination of (i) and (ii).

The visual examination of each test specimen is performed individually by 3 operators. The results are commonly reviewed by all the operators and when divergences in the results occur, a discussion leads to a given rating. It is thus a "laboratory result" by opposition to a result given by an individual.

From that, and to be able to comply with the specifications of the ISO 17025 [1], an inter-operator test on a reference set of samples is done regularly according to ISO 5725-2 [10]. Even if it is not an uncertainty estimation of a laboratory result, it is a basis for operator qualification.

6 Discussion

This test standard aims to give the following information: what is the preventive protection level against termites of a pine sapwood wood block containing a certain amount of product (retention) at a given concentration.

On one hand, for a fixed concentration, the retention of the wood preservative product in each sample depends on the initial weight and the volume. This is the reason why the wood selection is a crucial step, and the test gauge, despite its simplicity, became an essential tool. The retention of the wood product, considered as an intermediary result in the test process, has to be given in the test report. The uncertainty on the retention result was rather easily estimated according to ENV 13005 [11].

On the other hand, the final test result on the level of attack cannot be considered the same way as the retention result. In this very case, the result relies very much on the technical competence of the laboratory operators. Inter-operator comparisons according to ISO 5725-2 [10] were the only way to qualify the staff and improve the reliability of the test result based on visual rating of wood blocks. The laboratory staff was considered, to some extent, as a measuring material.

From a metrological point of view, methods such as 3D-image analysis could be of interest to become a substitute to the visual rating.

From a scientific point of view, a complete new way of measuring termite degradation should be developed. The EN 117 [4] test guidelines have been established as a standard many years ago for wood protection products that are no more on the market. This test standard has a scientific reality only for wood preservative formulations that combine toxicity and repellency. When the product is only toxic and not repellent, the termites have to ingest some treated wood before they die, leading then to an over-estimated level of attack when compared to field testing of the same product. This has been taken into consideration by the CEN/TC 38 "Durability of wood and wood-based products" and inter-laboratory cross-checking tests are on-going to develop a new method based on a choice test.

7 Conclusion

The expertise of the wood preservation laboratory is now recognized by the international scientific community and professional stakeholders in the sector. The accreditation is a firm guarantee of the laboratory's expertise for research partners or to respond to tenders for European projects.

The development of measuring instruments adapted to the constraints of our testing has strengthened the competence of operators and appears as an important factor in improving laboratory practices.

The metrological approach is so a long-term process. Extension of this approach to other standardized tests and the development of new methods are planned so as to take advantage of the experience already acquired in the development and adaptation of experimental methods and prenormative research.

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