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Using glycerol esters to prevent microbial growth on sunflower-based insulation panels

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In the indoor environment, the growth of microorganisms on building materials leads to the deterioration of both the materials and indoor air quality. As bio-based building materials usually contain cellulose or derivatives, they are likely to be much more sensitive to such degradation. Using glycerol esters could be a way to protect bio-based materials from microorganisms. Indeed, previous studies have highlighted the significant antimicrobial effect of glycerol esters and they are commonly used in the food industry as antimicrobial agents. In addition, as glycerol is a valuable by-product of the agroindustry, it would be an eco-friendly alternative, consistent with human health, to the classic ways of protecting bio-based materials against microorganisms. This study is part of a project that aims to (a) assess the hygrothermal performances and reaction to fire of sunflower panels and (b) study the antimicrobial efficiency of glycerol esters for the protection of such bio-based materials from microbial proliferation. The materials presented physical, thermal and hygroscopic properties similar to those of bio-based materials such as wood or hemp, encouraging their use as insulation materials. In addition, the glycerol esters showed significant antimicrobial effects but also a susceptibility to flammability. One unanticipated finding was that the untreated insulation material made of sunflower pith was classified as non-flammable.

Notation

t_i	ignition time (1 s after the appearance of the flame, must be greater than 5 s to be considered effective)
KT	total duration of effective combustion (total duration of the presence of flames exceeding the mark)
q	classification index
Σh	sum of the length of flames exceeding a particular mark

1. Introduction

Under certain hygro-metric conditions, building materials can become major targets of microbial growth (Verdier *et al.*, 2014). The proliferation of microbial growth leads to degradation of materials and may lead to a deterioration of air quality due to aerial particles (spores, fungal fragments, toxins etc.) and volatile organic compounds produced by these microorganisms (Verdier *et al.*, 2016). For several years, the World Health Organization and the French Observatory of Indoor Air Quality have been raising awareness of different types of indoor air pollution, including pollution of biological origin (CSHPF, 2006; WHO, 2009). These two degradation issues (materials and indoor air) are all the more sensitive with bio-sourced materials, which contain a significant proportion

of cellulose and/or derivatives that are nutrient sources promoting microbial growth. Until 2011, cellulose-based insulation materials were treated with boron salts for their flame-retardant and antimicrobial properties. A recent European Union directive 2009/94/EC bans the use of boric acid and salts as biocidal agents due to their category 2 classification as reprotoxic agents (EC, 2009). Today, the use of boron salts as flame retardants is still possible, if they are used without any biocidal claims. However, tolerated use is problematic from the point of view of health. Substitutes with reduced environmental and health impact should thus be developed. Recent works were carried out at Laboratoire Matériaux et Durabilité des Constructions and Laboratoire de Chimie Agro-industrielle (LCA) to develop bio-based products based on certain glycerol esters that have shown significant antimicrobial effects (Mikhailenko, 2015; Verdier *et al.*, 2017). Glycerol esters have specific surface-active properties that favour their use in many fields (cosmetics, food, pharmaceuticals, textiles etc.). They are made of a fatty acid connected to a glycerol molecule by an ester bond. The antimicrobial properties of glycerol esters have been studied for several years now and their disinfecting efficacy has been observed on a wide variety of microorganisms, including bacteria, yeasts, fungi and viruses (Desbois and Smith, 2009).

These antimicrobial, microbiocidal and/or growth-inhibiting properties are mainly governed by the structure and shape of the molecules – that is, the length of the carbon chain and the presence of double bonds (saturated/unsaturated). The literature reveals a strong antimicrobial potential of these molecules but, to the authors' knowledge, there is still no application to protect building materials. The use of this type of molecule is an innovative and environmentally friendly solution that could be used to protect bio-based insulation materials from microbial growth.

This paper presents the results from a project that aimed to develop bio-based insulation materials made of sunflower stalks, a by-product of sunflower cultivation, and glycerol esters to prevent microbial growth. The main issues addressed in this study were: (a) evaluation of the thermal and hygroscopic properties of two types of sunflower-based insulation materials, (b) investigation of the antimicrobial properties of glycerol esters and (c) evaluation of the impact of glycerol esters on the hygroscopic properties and reaction to fire of sunflower materials. The first section of the paper examines the basic properties of sunflower-based insulation materials, such as apparent density, thermal conductivity, water vapour sorption and permeability. Antimicrobial properties of glycerol esters will then be evaluated. Finally, the impact of glycerol esters on the water vapour sorption capacity and the reaction to fire of insulation materials will be investigated.

2. Materials and methods

2.1 Insulation using sunflower-based materials

Insulation materials were designed with sunflower stalks. A mechanical process developed by the Agromat technological transfer hall of LCA was carried out to separate pith from bark. On the basis of earlier studies (Evon *et al.*, 2015; Uitterhaegen *et al.*, 2019), a 'light' insulation material (density of about 50 kg/m³) was cast using pith (< 2.5 mm dia.) and a 'denser' one was cast using extrusion-refined bark (density of about 250 kg/m³). Samples were formulated with water and a starchy binder, then compression moulded at ambient temperature in different metallic moulds. The proportions are presented in Table 1. The casting protocol was as follows for both materials.

- Manual mixing of sunflower aggregates and binder in the dry state.
- Incorporation of warm water (60–70°C) and continuation of mixing until the water was homogeneously distributed in the mixture.
- Incorporation of the mixture inside a mould (with baking paper placed on the bottom of the mould and on the surface of the piston to prevent the mixture from sticking to the mould).

Table 1. Mix proportions of insulation materials

Material	Aggregates: % dry mass	Binder: % dry mass	Binder dilution: %
Pith	90.9	9.1	2.5
Bark	87	13	4

- Compaction with a piston under cold hydraulic press for 90 s per sample. The compaction was controlled with a target density for each sample and stopped when the press came into contact with the mould riser.
- Removal of the sample from the mould and storage inside a ventilated oven (40°C) until mass stabilisation. In order to limit the deformation of sample due to drying, especially the plate samples (5 mm thick), a metal grid was placed over the samples.

2.2 Glycerol esters – XUG

The glycerol esters used in this study were derivatives of biodiesel production (Mikhailenko, 2015) supplied by Kemerid. The product used in this paper, referred to as XUG, consisted of glyceryl mono undecylenate (50–60%), glyceryl di undecylenate (35–45%), glyceryl tri undecylenate (1–5%) and undecylenic acid (UA). As the latter is likely to present a significant antibacterial activity, the decision was made to test two types of XUG: a 'washed' XUG, containing about 0.5–2% of UA and an 'unwashed' XUG, containing 10–15% UA. Several concentrations of XUG were diluted with water and sprayed on the sample using a trigger spray bottle. The quantity was controlled by mass measurement. As XUG presents surfactant properties, its viscosity is temperature dependent. In order to carry out a repeatable covering method, the XUG and samples were conditioned in an air-conditioned room at 23 ± 1.5°C before each application.

2.3 Thermal conductivity

Thermal conductivity was evaluated by the guarded hot-plate method. Measurements were carried out with a λ -Meter EP500 at 23°C, with a difference of 10°C between the plates. Before each test, samples were conditioned at 5% relative humidity (RH) until mass stabilisation. The samples were wrapped in cellophane to prevent water migration during the test.

The presented results are the average of four measurements on different rectangular prisms (15 cm × 15 cm × 5 cm).

2.4 Sorption isotherms (dynamic vapour sorption (DVS))

The sorption isotherms of the materials were evaluated by the DVS method, previously described in Bui *et al.* (2017), Cagnon *et al.* (2014) and Laborel-Préneron *et al.* (2018). For

each test, five samples were tested in order to obtain representative results of the material. The mass was 0.15 ± 0.05 g (~ 3.4 cm³) and the duration of the test was between 4 and 5 days. When the mass was 24.0 ± 4.0 mg (~ 0.55 cm³), the duration of the test was between 2 and 3 days. Before testing, the samples were dried at 50°C in the DVS apparatus for 3 h. The tests were carried out at 23°C. The RH was regulated in successive stages from 0 to 95% in steps of 20% (15% for the last step). The mass stabilisation criterion (dm/dt) for the next step was chosen to be $\leq 5 \times 10^{-4}$ /min over a 10 min period (Cagnon *et al.*, 2014). The water content was calculated as the ratio of the mass of water contained in the sample to the mass of the dry sample (at RH \sim 0%). The test samples were uncoated (control), one-side coated (XUG-partial) or fully coated (XUG-full).

2.5 Water vapour permeability

The water vapour transmission properties were investigated on pith and bark samples according to ISO 12 572 (AFNOR, 2016). The wet cup test was used to investigate the performance in high-humidity conditions. Assuming a low resistance to water vapour, the adopted conditions were 23°C and 50/93% RH. A supersaturated solution of potassium nitrate was used in the cup to regulate the RH at 93%. Samples were sealed in the cup, over saline, with a wax–paraffin mixture. The test assemblies (sample + saline + cup) were placed in the test chamber, in which the RH was regulated at 50% with a supersaturated solution of sodium dichromate. Inside the chamber, an air flow ($v > 2$ m/s) parallel to the surface of samples was set up using a small fan (12 V). The room temperature was maintained at 23°C by an air-conditioning system.

The resistance of the materials to water vapour diffusion was expressed by the water vapour resistance factor (μ), derived from the water vapour permeability, and was calculated according to ISO 12 572. The presented results are the average of five or six samples in two separate chambers, for each type of material.

2.6 Reaction to fire

The reaction-to-fire test was carried out as a radiation test, according to NFP 92-501 (AFNOR, 1995). Six samples of 150 mm \times 150 mm were glued (Metylan Ovalit) side by side on a non-flammable gypsum board in order to reach the size requirement for the test (400 mm \times 300 mm). The test consists of submitting the sample to the action of a radiating heat source (500 W). When the material ignites, the heating source is moved away. Several parameters were noted during the test: t_i , the ignition time (1 s after the appearance of the flame, must be greater than 5 s to be considered effective); Σh , the sum of the lengths of flames exceeding a particular mark; KT , the total duration of effective combustion (total duration of

Table 2. q index and corresponding classification to fire, according to NFP 92-507 (AFNOR, 2004)

q index	Classification	Description
$q < 2.5$	M1	Non-flammable
$2.5 < q < 15$	M2	Hardly flammable
$15 < q < 50$	M3	Moderately flammable
$q \geq 50$	M4	Easily flammable

the presence of flames exceeding the mark). These parameters were then used to calculate the classification index q and deduce the classification to fire (Table 2). The index q was calculated according to:

$$q = \frac{100 \sum h}{t_i \sqrt{\Delta T}}$$

2.7 Inhibitory concentration test

To evaluate the optimal concentration of XUG to protect materials from microbial proliferation, a growth test was carried out on two fungal strains. As fatty acids also show antimicrobial properties (Bergsson *et al.*, 2002; Shi *et al.*, 2016; Valentin *et al.*, 2012), tests were carried out on three products: UA, unwashed XUG (XUG + UA) and washed XUG. The fungal strains used in this study were part of the FCBA Bordeaux collection. The strains *Aspergillus niger*, commonly identified on indoor building materials (Verdier *et al.*, 2014), and *Trichoderma vinide*, known to be more resistant to chemical treatment, were propagated on malt/agar (4%:2%) plates incubated at 22°C and 70% RH. The test was carried out on malt/agar medium supplemented prior solidification with washed XUG, unwashed XUG or UA at several concentrations. One plug of each strain was put on supplemented medium to evaluate the ability of each molecule to affect fungal growth. Experiments were carried out in three independent biological repeats. Agar plates were incubated at 22°C and 70% RH until complete growth. Radial growth was evaluated regularly during the test.

2.8 Resistance to natural proliferation

The goal of this experiment was to investigate the performance of coated XUG to protect materials from natural microbial growth. Bark samples were cut into specimens of size 5 cm \times 5 cm. An aqueous suspension containing 20% XUG was sprayed onto the surface of five samples (one side only) and five uncoated samples were used as the control. All samples were stored in a hermetic box containing a saturated solution of potassium nitrate in order to regulate the RH at 93%. The test was carried out at room temperature (23°C) for 4 weeks. Microbial growth was visually assessed once a week

Table 3. Rating scale for the assessment of mould (based on the work of Johansson *et al.* (2012))

Rating	Description of growth
0	No mould growth visible to the naked eye
1	Initial growth, sparse but clearly established growth
2	Scattered growth
3	Heavy growth, over more or less the entire surface

according to the rating scale shown in Table 3. This scale was adapted from Johansson *et al.* (2012).

3. Results and discussion

3.1 Basic properties of sunflower-based insulating materials

3.1.1 Apparent density

The apparent densities of samples after mass stabilisation in ambient air (21°C, RH~50%) are presented in Table 4. Pith- and bark-based prismatic samples are visible in Figures 1 and 2. Regarding the bark samples, the density of cylindrical samples was significantly lower than that of rectangular prisms. A possible explanation for this might be that the surface area of the rectangular prisms was different from that of the cylindrical samples, 225 cm² against 96 cm², respectively. This could generate more friction of bark particles against the wall of the cylindrical mould compared with the rectangular prisms, which may lead to less good stacking of the particles, thus inducing a lower density. Another possible explanation might be a more

Table 4. Apparent densities of samples (mean ± standard deviation)

Material	Type of sample	Density
Pith	Rectangular prism	44.1 ± 0.1
	Plate	44.1 ± 0.1
	Cylindrical	47.3 ± 1.9
Bark	Rectangular prism	232.5 ± 2.6
	Plate	261.9 ± 13.7
	Cylindrical	218.5 ± 5.7

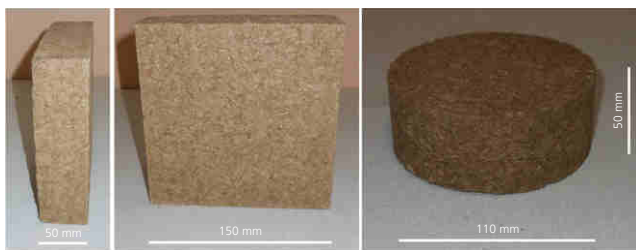


Figure 1. Photographs of bark samples



Figure 2. Photographs of pith samples

significant ‘expansion’ of cylindrical samples during drying, likely due to their size and cylindrical shape.

3.1.2 Thermal conductivity

A comparison of experimental thermal conductivities of the pith and bark samples and other insulation materials taken from the literature is shown in Figure 3. The thermal conductivity of the pith samples 0.038 ± 0.0002 W/(m.K) was similar to those of stone wool and expanded polystyrene, highlighting their possible use as insulation material. The thermal conductivity of the bark samples 0.054 ± 0.002 W/(m.K) was higher than that of the other materials, consistent with its higher density, but still in the range of common insulation materials.

3.1.3 Water vapour permeability

Figure 4 presents the values of the water vapour resistance factor (μ) derived from the water vapour permeability (ISO 12572 (ISO, 2016), wet cup method). The values calculated for pith and bark samples were compared with the resistance factors of different bio-based insulation materials

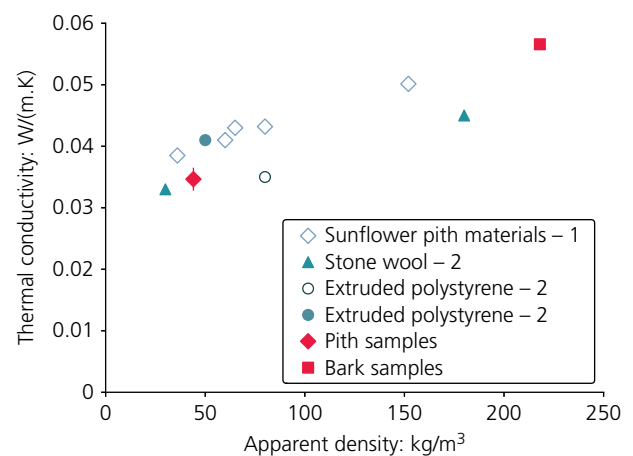


Figure 3. Thermal conductivity of pith samples and comparable insulation materials from the literature measured around 21–25°C. 1, Vandenbossche *et al.* (2012); 2, Papadopoulos (2005)

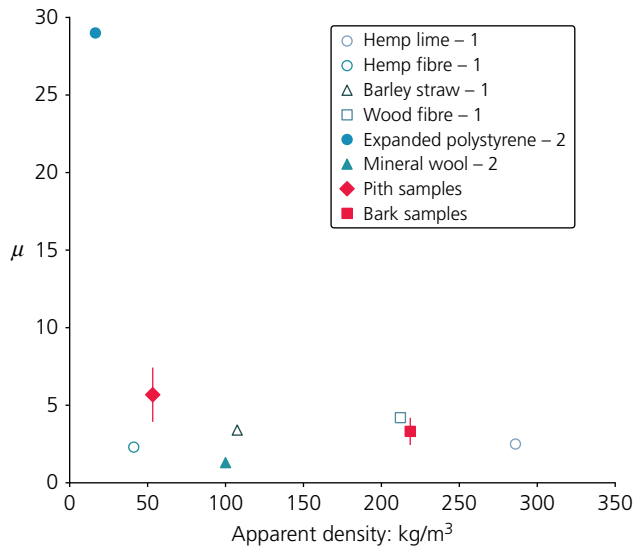


Figure 4. Water vapour resistance factor (μ) of pith and bark samples and other biobased insulation materials measured with the wet cup method. 1, Palumbo *et al.* (2016); 2, Jerman and Cerný (2012)

(Palumbo *et al.*, 2016) and expanded polystyrene (Jerman and Cerný, 2012), commonly used as insulation in buildings. The water vapour resistances of bark and pith samples were close to those of other bio-based insulation materials, with a slightly higher value for the pith sample. One can clearly see that the resistance factors of the bio-based materials were about six times lower than that of expanded polystyrene, reflecting their high permeability. These results are a good indicator for the use of sunflower as a vapour open insulation system in renovated buildings (e.g. Scheffler, 2011).

3.1.4 Water vapour sorption

The sorption isotherms of the pith and bark samples are presented in Figure 5. The curves are typical of isotherms obtained with a macroporous adsorbent, corresponding to a type II isotherm according to the International Union of Pure and Applied Chemistry classification (Sing, 1982). The isotherm represents unrestricted monolayer–multilayer adsorption. A strong increase in water content was observed at high RH, likely due to capillary condensation (Sing, 1982). The curve of the pith sample is close to the sorption isotherms of sunflower pith bulk particles (dashed curve) measured by Magniont (2010) with the DVS method. At 95% RH, the water content was near 40% of the dry mass for both samples, confirming a high water sorption capacity. As shown, the density of the sunflower material was quite low (about 50 kg/m³ for pith and 250 kg/m³ for bark), so was the volume fraction of adsorbed water. An implication of this is that thermal properties should be less impacted by water

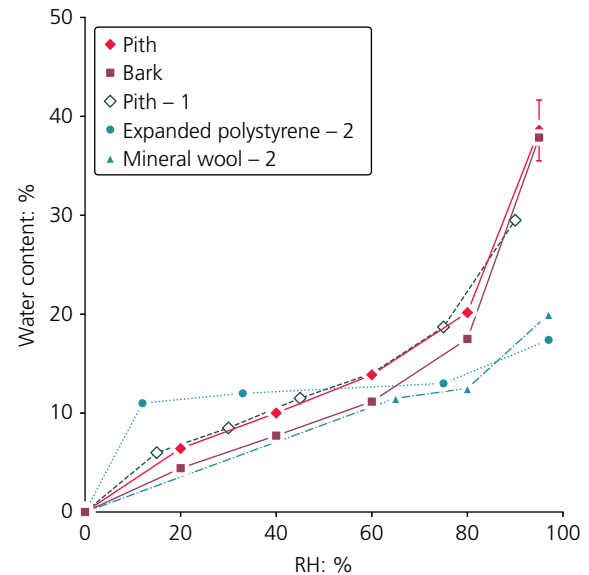


Figure 5. Sorption isotherm of pith and bark samples. Mean \pm 2 standard deviations ($n=5$). 1, Magniont (2010); 2, Jerman and Cerný (2012)

variation than expected, as shown in Figure 5. For comparison, Figure 5 also shows the sorption isotherms of two common insulation materials: mineral wool and expanded polystyrene (Jerman and Cerný, 2012). Expanded polystyrene tends to adsorb more water at low RH (under 50%) and less at high RH (over 50%) than the sunflower materials. Regarding mineral wool, its sorption capacity is similar to that of bark samples from 0% RH to 60% RH. Over 60% RH, both conventional materials showed lower sorption capacity than the sunflower materials.

3.2 Evaluation of the antimicrobial reactions of glycerol esters

Figure 6 shows the growth of *T. vinide* and *A. niger* in contact with UA, unwashed XUG or washed XUG. It should be noted that these tests were carried out in order to identify the most effective concentrations in a relatively short period of time. This is why tests were carried out on nutrient media that promote microbial growth. They do not reflect any antimicrobial activity that the products could have on an insulating material in real-world conditions.

As shown in Figures 6(a) and 6(d), 5% UA was sufficient to inhibit the growth of *A. niger* whereas 10% was not enough to inhibit the growth of *T. vinide*. The results obtained from unwashed XUG (Figures 6(b) and 6(e)) show that 5% was sufficient to inhibit the growth of *A. niger* and 20% was sufficient to inhibit the growth of *T. vinide*. Regarding washed

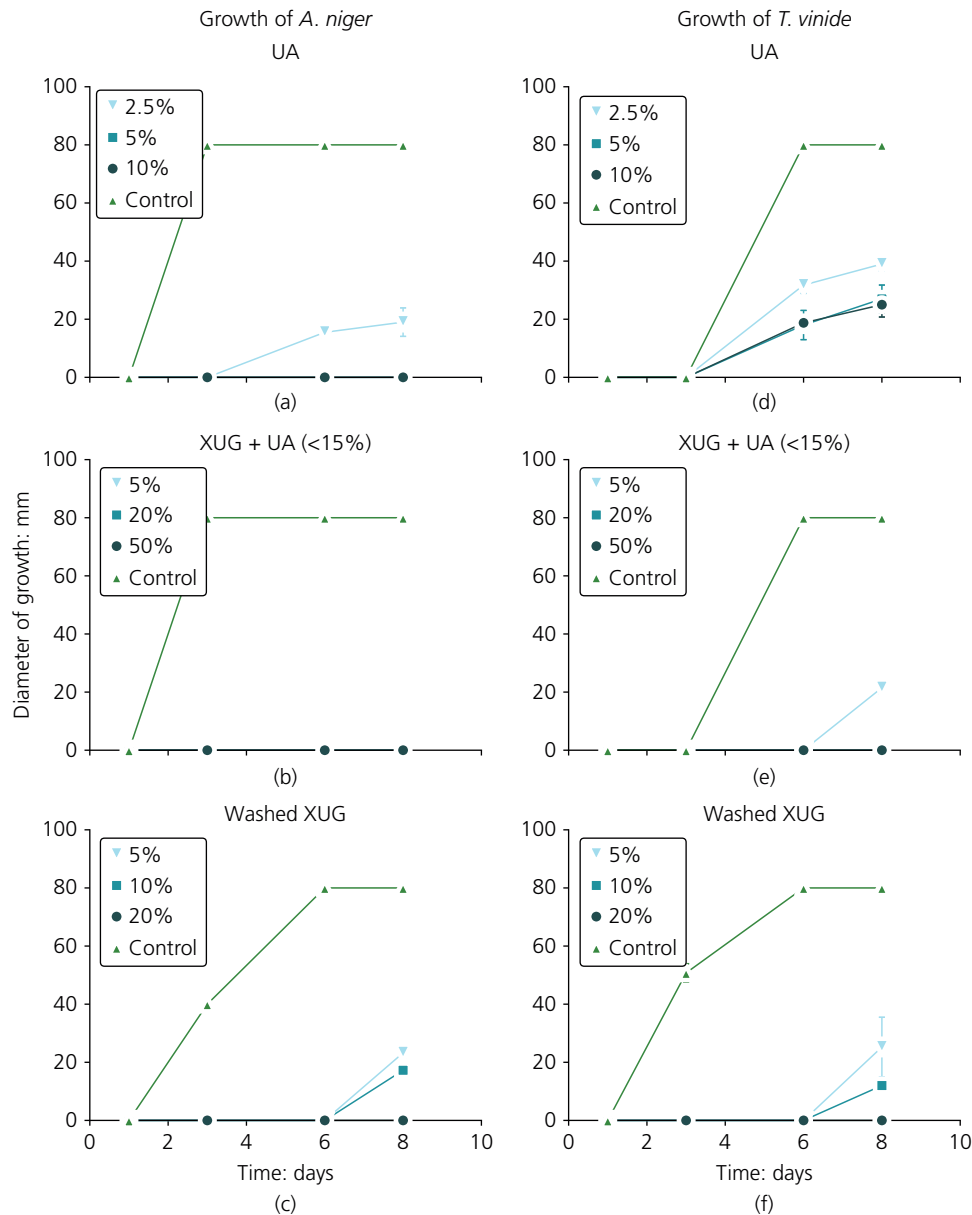


Figure 6. Results of the inhibitory concentration test of different products on *A. niger* (a–c) and *T. vinide* (d–f)

XUG (Figures 6(c) and 6(f)) at 5 and 10%, growth was limited for 6 days for both *A. niger* and *T. vinide*. Both fungi showed growth again after 8 days. A concentration of 20% washed XUG seems sufficient to inhibit growth for 8 days. These results show the synergic effect of XUG and UA, as the best inhibition performances were observed with 5 and 20% of the mix on *A. niger* and *T. vinide*, respectively. Surprisingly, UA was more efficient on *A. niger* than washed XUG and, conversely, washed XUG was more efficient on *T. vinide* than UA. These findings suggest that their antimicrobial actions are governed by different mechanisms.

An additional test was carried out on bark samples covered with XUG (20%), as described in Section 2.8. To favour the natural proliferation of microorganisms, samples were stored in a chamber at high RH (93%) for several days. Figure 7 compares microbial growth on the bark samples alone and samples coated with XUG (naturally contaminated).

After 26 days of incubation, both the uncoated and coated samples presented heavy growth over more or less the entire surface (level 3). In addition, it can be seen that growth on the uncoated samples was initiated during the first week as against

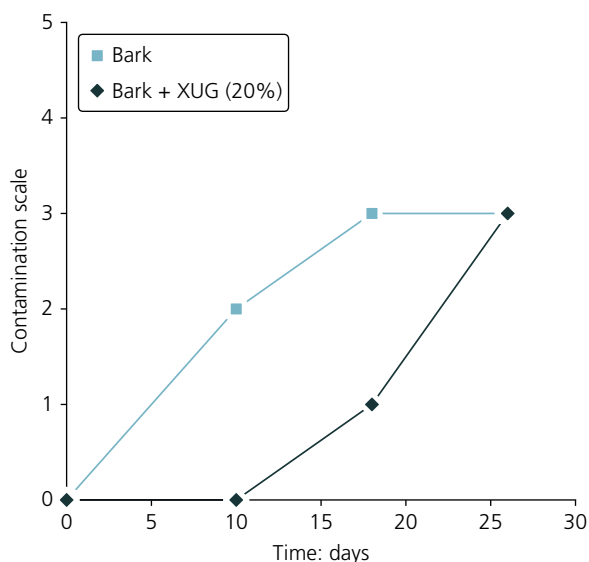


Figure 7. Evolution of microbial growth on naturally contaminated insulated bark samples

before the 10th day on the coated samples. Under the hygro-metric conditions of the test, the XUG did not prevent microbial growth but had a significant delaying effect. These results are consistent with data obtained from the inhibitory concentration test (Figure 6) and from a previous study (Verdier *et al.*, 2017) and emphasise the potential of this product for antimicrobial purposes.

Both the pith and bark insulation samples showed valuable thermal and hygroscopic properties and the XUG molecules showed significant antimicrobial effects. It is important to note that the intake of XUG did not interfere with some of the properties of the materials, in particular adsorption capacity and reaction to fire.

3.3 Impact of glycerol esters on water vapour sorption capacity and reaction to fire of sunflower-based insulation materials

3.3.1 Water vapour sorption

The sorption isotherms of pith and bark samples coated or not coated with XUG (20%) are shown in Figures 8 and 9. For the pith samples (Figure 8), it can be seen that there was no significant difference between pith partially covered and pith totally covered with XUG. In addition, the intake of XUG on samples seemed to lower water contents by about 5% from 20 to 80% RH. However, as the dry mass used to calculate water content was increased with the intake of XUG, one can assume that the observed decrease of water adsorption was 'artificial', due to an increase in the denominator based on

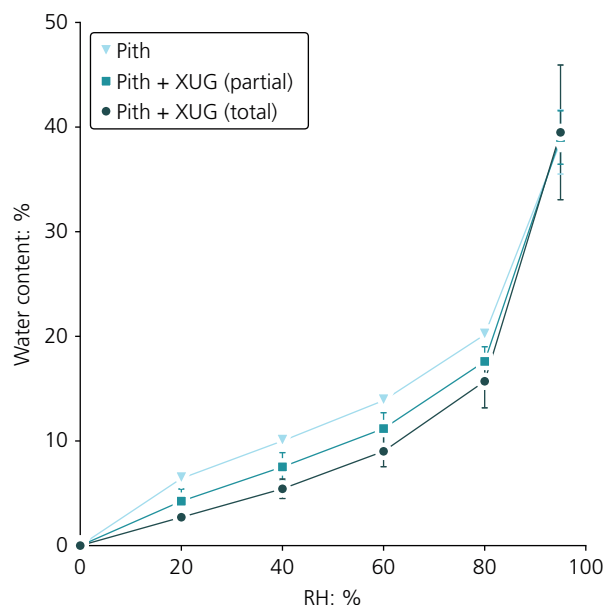


Figure 8. Sorption isotherm of pith samples. Mean ± 2 standard deviations ($n = 5$)

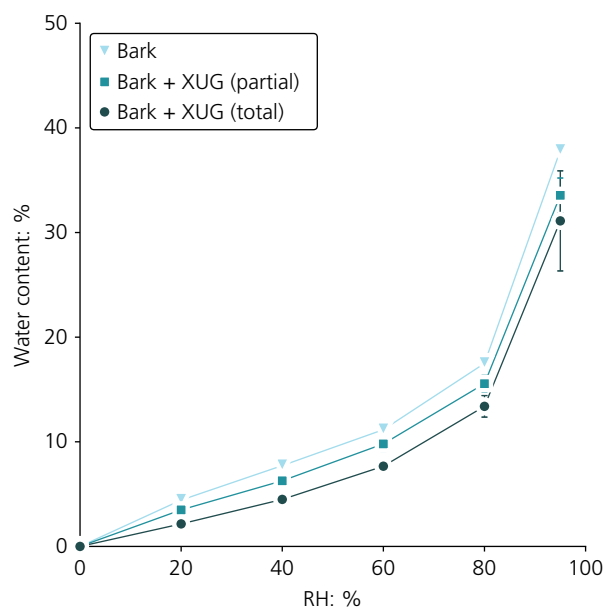


Figure 9. Sorption isotherm of bark samples. Mean ± 2 standard deviations ($n = 5$)

the calculations conducted. In any case, at 95% RH, the water content was similar to that of uncoated samples.

Similar results can be observed for the bark samples (Figure 9) and a similar conclusion can be drawn. Finally, for both the

bark and pith samples, one can assume that the impact of XUG on water sorption is negligible with regard to the high hygroscopic capacity of materials.

3.3.2 Reaction to fire

The results of the radiation tests are presented in Table 5.

The results show a difference in behaviour between the pith and bark samples. The pith had a M1 classification (non-flammable) whereas the bark had a M4 classification (easily flammable). In both cases, the addition of XUG induced an increase in the lengths of flames and, for the pith samples, an increase in the duration of actual combustion. It is therefore reasonable to say that XUG degrades the reaction to fire of sunflower-based materials. These results are consistent with the results obtained by Simon *et al.* (2008). The objective of that study was to develop innovative wood treatments based on functionalised oils, to give them properties in terms of fire resistance in particular. The results showed that the oils developed could instead degrade the reaction to fire.

Interestingly, insulation samples made of sunflower pith were classified as M1 (non-flammable). This difference with sunflower bark can be explained in part by their chemical composition, as this is a factor that impacts the reaction to fire (Palumbo Fernández, 2015). It can be seen from the data in Table 6 that sunflower bark is made up of a significantly higher proportion of hemicellulose than the pith (30% and 9%, respectively) and a lower proportion of ash (9% and 20%, respectively). These two factors will reduce flammability (Palumbo Fernández, 2015).

Table 5. Results of radiation test and corresponding classification of insulation materials

	Pith	Pith + XUG	Bark	Bark + XUG
Ignition time: s	13	5	20	11
Length of flames: cm	0	123	562	646
Duration of actual combustion: s	9	140	1180	1189
q index	0	207.91	81.8	170.31
Classification	M1	M4	M4	M4

Table 6. Main components of sunflower pith (Yin *et al.*, 2007) and bark (Khristova *et al.*, 1996)

	Pith	Bark
Cellulose: %	47	41
Hemicellulose: %	9	30
Pectins: %	6	—
Lignins: %	3	18
Ash: %	20	9

In accordance with the current results, previous studies have observed that bio-based insulation materials presented a better reaction to fire than commercial organic foams (polystyrene and polyurethane) and expanded polystyrene (Palumbo Fernández, 2015; SB&WRC, 2019).

4. Conclusions

The main goal of this project was to study the possibility of using sunflower stalks, a by-product of sunflower cultivation, and glycerol esters to produce an insulating product capable of resisting microbial growth. For that purpose, the thermal and hygroscopic properties of two types of sunflower-based insulation materials and the antimicrobial effects of glycerol esters were investigated. The impact of glycerol esters on sorption capacity and fire reaction of materials was also investigated.

Two types of insulation were studied: a light insulation made of sunflower pith (50 kg/m³) and a dense insulation made of sunflower bark (250 kg/m³). The samples were cast using a compaction method with a cold hydraulic press and their hygro-thermal properties were evaluated by measuring thermal conductivity, sorption capacity and water vapour permeability. Thermal conductivities of 34.7 mW/(m.K) for pith and 56.6 mW/(m.K) for bark were comparable with those of stone wool and expanded polystyrene, highlighting their potential for use as insulation materials. Moreover, a high sorption capacity and high permeability were observed for both materials. These results encourage the use of sunflower as a vapour open insulation in buildings to regulate indoor humidity variation and heat transfer.

Regarding the antimicrobial properties, different efficiencies were observed according to the tested molecules (UA and XUG) and organisms (*A. niger* and *T. vinide*). The highest inhibitory effect was observed for the XUG containing UA on both *A. niger* and *T. vinide*. In addition, a significant delaying effect on microbial growth was observed on bark samples coated with XUG. These results support the antimicrobial performance of such molecules observed in previous work (Verdier *et al.*, 2017) and are encouraging with regard to the use of XUG as protection from microbial growth.

According to the conclusion made in the Section 3.3.1 on water adsorption, it can be assumed that, in this study, the hygroscopic properties of the pith and bark samples were not significantly impacted by the addition of XUG on their surfaces. However, XUG is known to have inherently surface-active properties. Therefore, if this product is included in the mass during the casting of materials, it can be assumed that the water permeability would be increased. However, this could also negatively impact the thermal conductivity of

such material. These aspects should be addressed in further studies.

The radiation tests showed that the bark samples were easily flammable and the addition of XUG increases the flammability of insulation materials. Nevertheless, an interesting result was that the pith sample was classified as a non-flammable material, underlining its potential for use as a building material.

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