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Keywords (separated by '-')	Cross-laminated timber - Controlled-atmosphere cone calorimeter - Oxygen vitiation - Gases - Aerosols	
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Effects of Oxygen Concentration on the Reaction to Fire of Cross-Laminated Timber in a Controlled-Atmosphere Cone Calorimeter

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Keywords: Cross-laminated timber, Controlled-atmosphere cone calorimeter, Oxygen vitiation, Gases, Aerosols

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40 1. Introduction

42 In recent years, there has been a great deal of interest in sustainable construction,
43 with the objective of developing energy-efficient buildings with a low carbon foot-
44 print. To fulfill this objective, renewable and/or recycled construction materials
45 are increasingly used, as wood-based materials. In the building field, Cross-Lami-
46 nated Timber (CLT) is widely used because of its structural properties. With low
47 weight and fast construction times, CLT challenges the use of traditional dense
48 materials like steel and concrete. However, like most organic-based materials,
49 CLT decomposes when exposed to fire. Thus, it is important to study its behavior
50 under different fire scenarios to implement suitable protection methods to ensure
51 the safety of people. Especially, knowledge of gas and aerosol emissions from the
52 thermal decomposition of CLT is paramount. Indeed, during a building fire, most
53 deaths are due to smoke release [1]. Smoke reduces visibility, causes impaired
54 vision and respiratory problems due to irritating and asphyxiating gases. Conse-
55 quently, this leads to impairment of escape and increases the time to escape and
56 the probability of injuries or death [2]. To ensure safe evacuation, the standard
57 ISO 13571 [3] subdivides the risks to people escaping a fire into the effects of heat,
58 asphyxiant and irritant gases and visual obscuration by smoke. Each component
59 is assessed separately and untenability is defined when one of the components
60 reaches a level that prevents escape. This underlines the need to assess the toxicity
61 of materials to have a better understanding of phenomena occurring during a fire
62 and to improve fire safety engineering. Toxicants production and yield depend on
63 the material composition and the fire conditions including oxygen concentration
64 and temperature. An under-ventilated or low oxygen level (vitiated) atmosphere
65 increases the production of dangerous gases for humans. Indeed, the oxygen level
66 quickly decreases until it no longer allows complete combustion. A dense, carbon
67 monoxide (CO) rich smoke as well as other toxic gases and non-toxic gases are
68 produced. A complete assessment of the thermal decomposition of materials with
69 gaseous emissions and aerosols production is crucial for fire safety.

70 To date, there are many bench-scale devices available but there is no interna-
71 tional agreement or standard on how to assess this toxicity. The main challenges
72 are to correlate results with real scale as well as to simulate and control different
73 fire scenarios. The Controlled-Atmosphere Cone Calorimeter (CACC) is a modi-
74 fied design from the Cone Calorimeter developed by V. Babrauskas and standard-
75 ized in ISO 5660-1 [4]. The standard cone calorimeter is widely used to assess the
76 reaction to fire of a material but as its design is open, it represents well-ventilated
77 conditions and it does not enable reduced oxygen atmospheres. Consequently,
78 research focused on the development of enclosed cone calorimeters where the oxy-
79 gen level of the atmosphere can be controlled. Werrel categorized them into two
80 designs: closed designs and open designs [5]. These latter have no connection
81 between the exhaust hood and the enclosure. Several open designs were set up to
82 study the reaction to fire of various materials, *e.g.* polymethylmethacrylate
83 (PMMA) [5–10], polyethylene (PE) [7, 11], polyisocyanurate (PUR) [12], acryloni-
84 trile butadiene styrene (ABS) [7, 13], polyvinyl chloride (PVC) [10, 14], sandwich

85 material [15] or wood [16]. As there was no international agreement on the CACC
86 design nor the experimental protocol, Marquis et al. [6] investigated the interpre-
87 tation and accuracy of results with different setup. They observed that the design
88 of the combustion enclosure may affect the material burning behavior and the
89 accuracy of measurement. Therefore, they suggested constructing a chimney
90 between the enclosure and the exhaust hood to reduce the post-oxidation phe-
91 nomenon. From this work, the CACC and the experimental protocol associated
92 are now standardized with ISO 5660-5:2020 [17]. In this standard, new equations
93 developed by Werrel et al. [5] to calculate the Heat Release Rate (HRR) are pre-
94 sented. Indeed, the exhaust gases are diluted by excess air drawn from the labora-
95 tory surroundings. The heat-induced changes in the dilution ratio affect the
96 measurement of the oxygen and the calculation of the HRR. The error in the
97 HRR calculation increases at a significant order of magnitude ($\approx 30\%$) when the
98 oxygen content in the enclosure is decreased below 18 vol % [5]. Thus, Werrel
99 et al. [5] considered the variations in the flow rate of the ambient air from the
100 laboratory drawn into the exhaust hood to avoid inaccurate HRR calculations.

101 Work done on several CACC designs and materials has shown trends in the
102 effect of oxygen. Indeed, the HRR [7, 8] and the Mass Loss Rate (MLR) [7, 8, 18]
103 decrease when oxygen concentration decreases (vitiation). On the contrary, the
104 carbon monoxide production [7, 19] increases with vitiation whereas the time to
105 ignition remain unchanged [8]. However, the effect of oxygen on the fire behavior
106 of CLT in a CACC has never been studied. More specifically, there is a lack of
107 data on the gaseous emissions and the aerosols production of CLT in vitiated
108 environments.

109 This paper depicts the results of the effects of oxygen concentration on the reac-
110 tion to fire and gas and aerosol production of CLT in a CACC. To have a better
111 understanding of the complex phenomena occurring, a homemade bench is
112 designed where a Fourier Transform Infrared (FTIR) spectrometer as well as an
113 Electrical Low Pressure Impactor (ELPI) are coupled to the CACC. Particularly,
114 the samplings for FTIR and ELPI analysis are made in the chimney instead of in
115 the exhaust duct in order to limit the loss of species due to dilution/oxidation with
116 ambient air. The experiments are realized at an irradiance of 50 and 20 kW/m² to
117 assess the fire risks of CLT under different fire scenarios, respectively well-venti-
118 lated fires and smoldering.

119 **2. Material and Method**

120 **2.1. Material**

121 The material used in this study is a commercial Cross-Laminated Timber (CLT)
122 made from spruce. The panels used consist of two 20 mm thick plies glued per-
123 pendicular to each other. The glue used is polyurethane based and it does not
124 release formaldehyde. The sample dimensions are 100 mm long, 100 mm wide and
125 40 mm thick. It has a specific gravity of 500 kg/m³, a thermal conductivity of 0.12
126 W/m.K and a heat capacity of 1600 J/kg K (Table 1).

Table 1
CLT Properties Given by the Manufacturer

Specific gravity	500 kg/m ³
Thermal conductivity	0.12 W/m K
Heat capacity	1600 J/kg K

127 The specimens are conditioned at 23 ± 2 °C and at a relative humidity of $50 \pm$
128 5% until the mass is constant, in order to reach a moisture of wood close to 11%
129 as described in the standard EN 13238 [20].

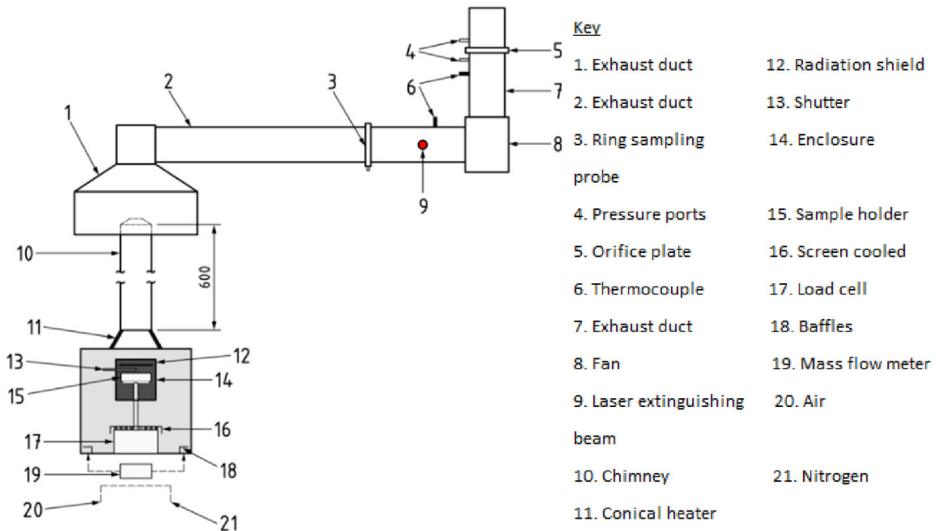
130 2.2. Controlled-Atmosphere Cone Calorimeter

131 Very recently an experimental standard was released, it is entitled “ISO 5660–5:
132 Heat release rate (cone calorimeter method) and smoke production rate (dynamic
133 measurement) under reduced oxygen atmospheres” [17]. It specifies how to use the
134 cone calorimeter to perform tests in controlled atmospheres. The device is able to
135 work under 1% to 21% of oxygen and to deliver between 10 and 180 L/min of
136 gas. The enclosure, described in Figure 1, is a stainless-steel box of 37 30 30 cm³ in
137 dimensions with the standard cone heater on top, a door and a window. Inside,
138 there is a load cell and two gas inlet ports. The atmosphere is adjusted by control
139 of the volume flow rate of air and nitrogen, each with a rotameter and monitored
140 by an additional oxygen analyzer that is linked to the enclosure. Moreover, to
141 avoid excessive heating and radiation of the system, a cooling ring is set between
142 the cone heater and the top of the enclosure as well as a cooling hood on the load
143 cell and a cooling serpentine around the load cell’s rod.

144 The experimental method is similar to that of the standard cone calorimeter.
145 The surface area of the sample is 100 100 mm². The sample can be exposed to a
146 constant irradiance level up to 100 kW/m². Above the sample, a spark plug ignites
147 the flammable gases that might be released. These gases are then collected in the
148 exhaust hood and send through a duct provided with thermocouples, pressure sen-
149 sor, smoke measurement and a ring probe for O₂, CO and CO₂ analyzers. As a
150 result, heat release rate, mass loss rate, smoke density and gas concentration are
151 assessed.

152 A drawback of the CACC is the possible post-oxidation of the fire effluent
153 between the enclosure and the exhaust sampling point that would modify HRR
154 and gas measurements. To overcome this issue, the standard specifies to set a
155 chimney of 60 cm long and of 11.5 cm in diameter above the cone heater. It can
156 be made of metal or of quartz; the differences have been discussed in [6].

157 In this study, the CACC depicted above is used but with some modifications.
158 Indeed, to avoid heat losses, the four walls of the enclosure are insulated with
159 25 mm calcium silicate panels. Moreover, a new metallic chimney is set to ensure
160 better sampling of effluents.

Effects of Oxygen Concentration on the Reaction to Fire of Cross-Laminated Timber**Figure 1. CACC design from ISO 5660-5 [16].****161 2.3. Controlled-Atmosphere Cone Calorimeter Coupled with FTIR and ELPI**

162 To study the influence of oxygen concentration on the gas production and the
 163 aerosol size distribution, two devices were respectively coupled to the CACC: a
 164 Fourier Transform Infrared spectrometer and an Electrical Low Pressure Impac-
 165 tor. Instead of classical sampling in the duct, the raw sampling method is used in
 166 this study. It was recently developed in order to avoid the loss of effluent when
 167 assessing its composition. Indeed, the effluents can condensate while being trans-
 168 ported and more importantly when diluted species are lost due to their low con-
 169 centration (acrolein or formaldehyde for example). Dilution lowers the dew point
 170 of the sample so that the water vapor in the effluents does not condense when the
 171 sample is cooled by dilution [21]. Consequently, the sampling of the effluent is
 172 made in the chimney instead of in the exhaust duct. Further details on the sam-
 173 pling method can be found in literature [10, 12, 21, 22].

174 In this study, a new metallic chimney (Figure 2a), was designed in order to
 175 insert a sampling probe for the FTIR and another one for the ELPI. The chimney
 176 is 60 cm long and of 11.5 cm in diameter with its top diameter reduced to 5 cm to
 177 assure a sufficient velocity flow of the smoke in the duct. The FTIR sampling
 178 probe (Figure 2b) is located near the outlet of the cone and is composed of two
 179 flute probes of four holes each to have a homogenous mixture. The holes face
 180 away the stream of effluent. The ELPI measurement consists in a single point
 181 probe that ensures isokinetic sampling.

182 An Antaris IGS FTIR spectrometer equipped with a nitrogen Mercury Cad-
 183 mium Telluride (MCT) detector, 0.2L volume gas cell, and a 2 m optical path
 184 length is set. Each scan was taken at 1.76 s and 0.5 cm^{-1} as spectral resolution. At
 185 the output of the chimney, the gases are transported through a PTFE line. The

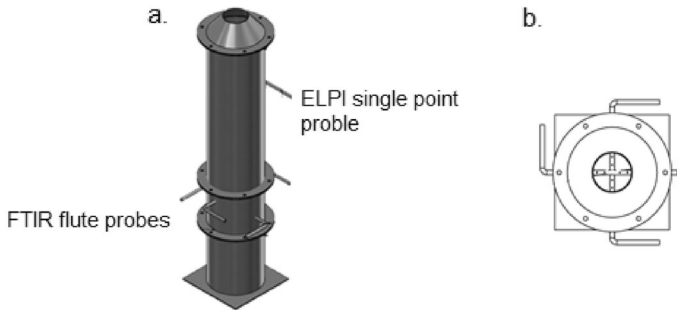


Figure 2. (a) designed metallic chimney for raw sampling; (b) top view of FTIR probes.

186 FTIR gas cell as well as the PTFE gas transport line are heated at 180 °C to avoid
 187 condensation. In the end, gases are cooled down, dried, filtered and then evacu-
 188 ated with a membrane pump. The gas cell was kept at a constant pressure of
 189 650 Torr with a pressure gauge with regulating valve. Each gas (CO₂, CO, H₂O,
 190 CH₄, CH₂O, C₂H₄O, C₂H₄, C₃H₆) is calibrated using the Classical Least Square
 191 (CLS) algorithm. For each gas, a dilution range was analyzed with the FTIR.
 192 Characteristic peaks on FTIR spectra were carefully selected by considering all the
 193 unavoidable interferences between substances [23].

194 ELPIs are cascade impactors widely used to measure in real-time the particle
 195 size distribution and the concentration of aerosols. An ELPI as well as two
 196 diluters from Dekati are set. The ELPI principle consists of three main steps.
 197 First, the sampled aerosols are subjected to a unipolar positive ion environment in
 198 a corona charger where they are electrically charged. Then, the charged particles
 199 enter a low-pressure cascade impactor where they are classified into size of 7 nm
 200 to 10 μm according to their aerodynamic diameter which determines their deposi-
 201 tion at a particular ELPI stage. Finally, the charges carried by the aerosols are
 202 continuously measured at each impactor stage by sensitive electrometers. The
 203 measured current values are converted to give a number of particles by cm³ using
 204 transfer functions provided by manufacturers. The transport line is an antistatic
 205 PTFE pipe heated at 180°C to avoid effects of condensed humidity on the mea-
 206 surement. A vacuum pump is set to ensure an isokinetic sampling flow at a rate of
 207 10 L/min. Double dilution of the effluents is set upstream the ELPI particle sizer
 208 with two diluters DI-1000 from Dekati. The first one is heated at 200°C to avoid
 209 loss in aerosol concentration while the second one prevents nucleation of the aero-
 210 sols. The diluters are set in series to provide a ratio of dilution of 83.

211 2.4. Experimental Procedure

212 Samples are tested in the horizontal orientation. The sample holder is used with a
 213 frame to reduce the exposed surface of the sample. A 64 kg/m³ ceramic fiber insu-
 214 lation pad is put on the backside of the sample as specified in the ISO 5660-1
 215 standard [4]. The volumetric flow rate of extraction through the exhaust duct of

216 the calorimeter is set at 24 ± 2 L/s at 23 °C. The inlet volume flow rate is set at
217 150 ± 5 L/min at 23 °C. Tests are carried out at 50 and 20 kW/m² and at 21, 18,
218 15 and 10 vol% of oxygen respectively. All tests are performed at least three times
219 and the curve presented represents a single specimen that best fits the mean curve.
220 Data were collected with a 1 s sampling interval for the cone measurement, 1 s for
221 the ELPI and 1.76 s for the FTIR.

222 3. Results and Discussion

223 The reaction to fire of Cross-Laminated Timber is strongly dependent on the envi-
224 ronment conditions such as the oxygen level and the heat flux. The combustion is
225 considered incomplete when the CO/CO₂ ratio is greater than 0.05 [25]. For the
226 whole study, the time to ignition is defined as t_i , the time to flameout as t_f , the
227 peak of HRR as pHRR and the peak of MLR as pMLR.

228 3.1. Influence of Oxygen Level at 50 kW/m²

229 3.1.1. Mass Loss and Heat Release 3.1.1.1. Mass Loss The influence of oxygen
230 level on the remaining mass and the mass loss rate at 50 kW/m² was first studied
231 (Figure 3). The decomposition process of CLT at 21 vol% O₂ is first described
232 before studying the effect of oxygen.

233 The decomposition pathway of CLT at 21 vol% O₂ is assessed via the mass loss
234 (Figure 3b). First, between 0 and 19 s, as the temperature at the surface of CLT
235 increases, the sample starts to degrade. The free water contained in wood (around
236 11%) evaporates. Simultaneously, the chemical bonds of wood polymer chains
237 (cellulose, hemicellulose and lignin) start to break [26, 27]. Consequently, tars and
238 decomposition gases are produced. As the gases leave the solid, they encounter the
239 oxygen present in the atmosphere. Concurrently, oxygen may diffuse into the solid
240 and oxidize the wood effluents. Triggered by the igniter, the mixture ignites and
241 the flame spreads onto the whole surface of the sample. It results, in a second
242 step, in a fast increase of the MLR, which reaches a maximum at 60 s. A car-
243 bonaceous layer is formed on top of the wood, which reduces the heat transfers
244 between the wood surface and the pyrolysis front. This carbonaceous layer creates
245 then a thermal insulation [28]. As a result, pyrolysis of wood slows down and the
246 MLR decreases. From 200 s, a small increase in MLR occurs. To understand this
247 behavior, five thermocouples were inserted in CLT at 2, 10, 20, 30 and 38 mm
248 from the exposed surface. When the MLR increased, the thermocouple inserted at
249 10 mm from the surface reached a 100 °C plateau (not shown). This temperature is
250 assigned to water evolution and suggests that CLT undergoes dehydration reac-
251 tions [29]. At longer times ($t > 200$ s), the sample reaches a thermal steady state,
252 which is characterized by a quasi-constant MLR (Figure 3b) and a constant thick-
253 ness of char layer [30]. In a next step, a second peak of MLR occurs around
254 2500 s. At this peak, the temperature measured at 38 mm from the surface of
255 CLT is 320 °C. It means that the pyrolysis front reaches the back of the sample
256 and thus the ceramic holder induces a thermal feedback (accumulation of heat)
257 that accelerates the decomposition [28]. Finally, flame extinguishes as the fuel con-

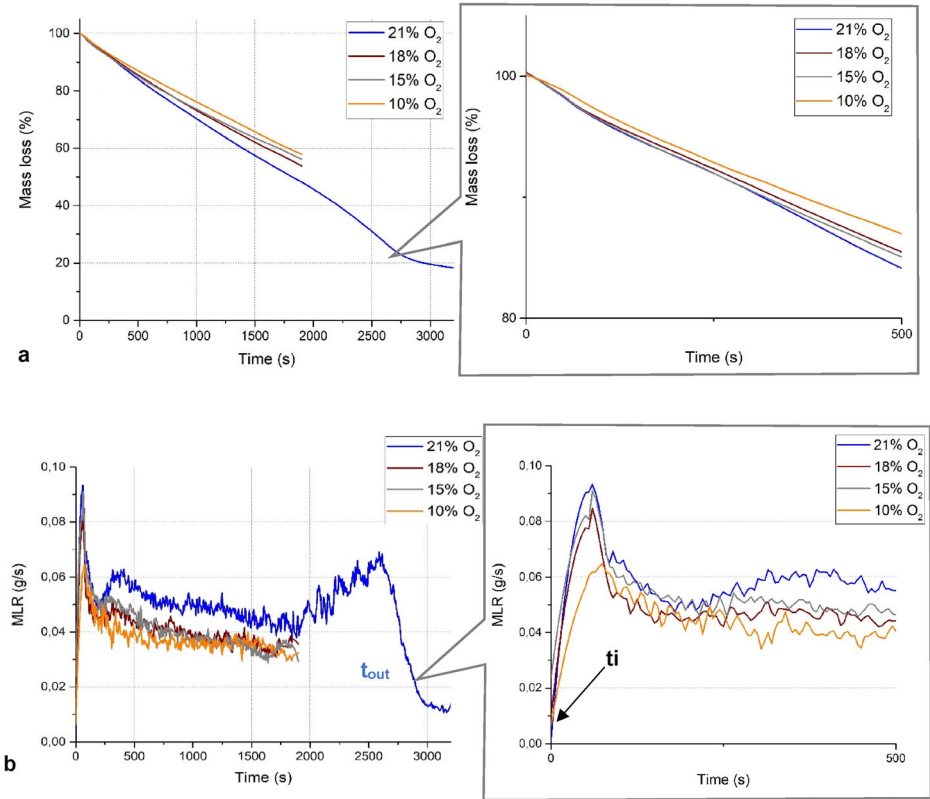


Figure 3. Effect of oxygen concentration on: (a) mass loss, (b) MLR.

258 tained in the solid phase diminishes and the MLR then decreases. Smoldering will
 259 then take place in the remaining char (visual observation).

260 Furthermore, the oxygen level affects the combustion process of CLT. Indeed,
 261 its decomposition occurs with flames at 21, 18 and 15 vol% O₂ but without flames
 262 at 10 vol% O₂. However, the MLR curves have all the same shape whatever the
 263 oxygen level (Figure 3b). The chemical reactions occurring in the condensed phase
 264 do not depend on the mass fraction of oxygen. Indeed, before 105 s, the mass loss
 265 curves and the peak of MLR (at ignition) at 21, 18 and 15 vol% O₂ (Figure 3a),
 266 (Table 2) are similar. This suggests that at the early stage of the test, the heat
 267 received at the surface of CLT mainly governs the decomposition of the sample.
 268 However, oxygen does modify the kinetics of decomposition, which are acceler-
 269 ated at high oxygen level. After 105 s, the mass loss of the specimen at 21 vol%
 270 O₂ accelerates while the mass losses at 18 and 15 vol% O₂ remain close with less
 271 than 0.7% of difference between them. Nevertheless, after 1100 s, the decomposi-
 272 tion becomes faster at 18 vol% O₂ (Figure 3a). Indeed, the decrease in oxygen
 273 level reduces the flame temperature and thus the flame thermal feedback to the
 274 surface of the solid [30]. Consequently, as less heat is transferred to the pyrolysis

Table 2
Influence of Oxygen on the Fire Parameters at 50 kW/m²

Oxygen level (%)	t _i (s)	pMLR (g/s)	pHRR (kW/m ²)
21	19	0.093	143
Standard error	1.5	0.004	12
18	18	0.085	138
Standard error	1.5	0.009	8
15	21	0.091	141
Standard error	0.7	0.010	9
10	–	0.065	41
Standard error		0.019	13

front with decreasing oxygen, the decomposition rate of wood is slower and the amount of CLT residue after 32 min is 48% at 21 vol% O₂ vs. 56% at 15 vol% O₂ (Figure 4) [27]. At 10 vol% O₂, the MLR curve has the same shape as other oxygen levels but the decomposition occurs without flames. Though the heat flux is high enough to decompose the sample into volatiles, there is not enough oxygen for a flaming combustion to occur. Thus, the pMLR is decreased by 43% compared to the pMLR at 21 vol% of O₂ (Table 2). As char is forming, the MLR slows down and reaches a quasi-steady state.

In the condensed phase, the oxygen concentration affects the combustion process (flaming or non-flaming) and the kinetics of decomposition. The higher the oxygen content, the faster the mass loss is.

3.1.1.2. Ignition and Heat Release In the gas phase, depicted here by the oxygen consumption based HRR, the reaction between the decomposition gases released from wood and the ambient air occurs near the surface of the sample. The mixture fuel/oxidant needs to reach the lean flammability limit to have a reaction of combustion. If there is enough energy produced by the reaction to overcome heat losses, the flame will spread on the whole surface of the sample [31]. For flaming combustion experiments (21, 18 and 15 vol% O₂), their times to ignition (Table 2) are not influenced by oxygen concentration as they are in the uncertainty range (± 3 s). Moreover, the HRR curves of flaming samples have all the same shape (Figure 5). Indeed, the HRR increases rapidly to reach a peak and decreases after as char forms. Then, the HRR curve reaches a steady state. As the pHRRs are in the same range considering the standard errors of pHRRs at 21, 18 and 15% O₂ (Table 2), the oxygen concentration does not seem to have a significant influence. The steady state reached after the peak is related to the thickness of the char layer which is almost constant [31]. With the decrease of oxygen concentration, the layer of char becomes thicker, involving less heat transferred to the pyrolysis front. As a result, at the steady state, the higher the oxygen level is, the greater the HRR is. Furthermore, at 15 vol% O₂, the weak concentration of oxygen induces a reduction in the flame intensity and thus the HRR decreases. At the end of the test, small flames are located on the edges of the specimen.

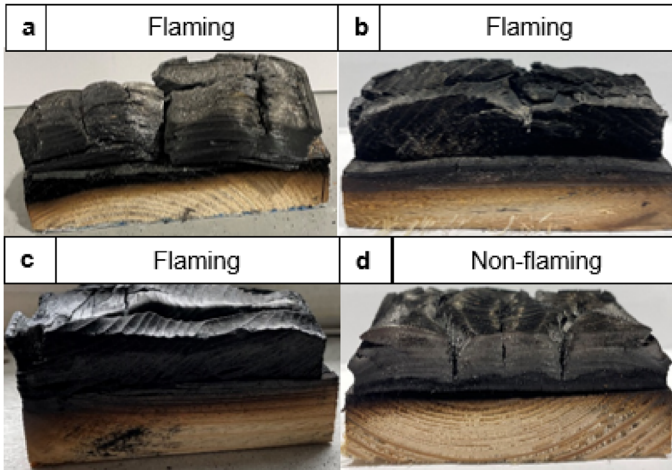


Figure 4. Sliced CLT after 32 min of test at 50 kW/m² at (a) 21% O₂, (b) 18% O₂, (c) 15% O₂ and (d) 10% O₂.

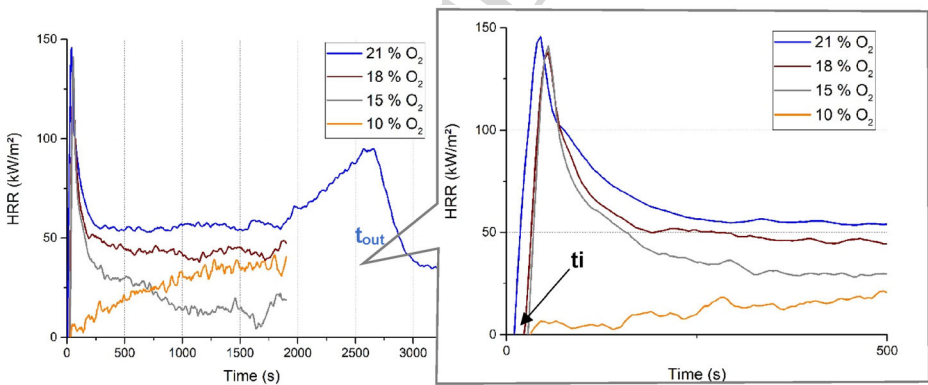


Figure 5. Effect of oxygen concentration on the HRR.

306 From 15 to 10 vol% O₂, the combustion switches to flaming to non-flaming
 307 mode (Figure 5). At 10 vol% O₂, the mixture fuel/oxidant remains below the
 308 flammability limit because of the lack of oxygen. Thus, the HRR is low in the
 309 early stage of the test but slowly increases next. Indeed, because of the heat
 310 received at the surface of the char, smoldering occurs. The HRR remains then
 311 steady at a level of 40 kW/m².

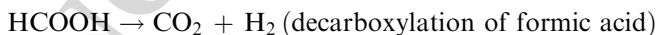
312 In summary, at 50 kW/m², two behaviors are observed according to the oxygen
 313 concentration: flaming for 21%, 18% and 15% of oxygen and non-flaming for
 314 10% of oxygen. The pMLR and times to ignition are not affected by oxygen
 315 depletion in flaming combustion. Additionally, the oxygen concentration does not
 316 affect the pHRRs values of flaming samples but it does influence the steady state
 317 reached then.

318 3.1.2. *Gas Production* In addition to influencing the fire parameters of the CLT,
319 the oxygen level also has a strong influence on the gas production. The nature of
320 gaseous products and their quantities depend on the oxygen concentration.

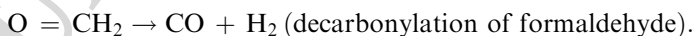
321 Wood combustion is a multi-component complex process as it is composed of
322 three polymers: cellulose, hemicellulose and lignin. In general, thermal degradation
323 of wood starts with the evaporation of water by dehydration reactions. Then, dif-
324 ferent chemical bonds within the polymers are broken following either depolymer-
325 ization (breaking of the bonds between the monomer units of the wood polymers),
326 fragmentation (linkage of covalent bonds of polymers) and/or char formation.
327 This results in the release of volatile compounds and rearrangement reactions
328 within the wood matrix. Some of the volatiles produced are unstable and may
329 undergo secondary reactions, known as cracking (breaking of chemical bonds
330 within the volatile compounds) or recombination (combination of volatiles com-
331 pounds) [32].

332 More precisely, for the flaming specimens (21, 18 and 15 vol% O₂), many con-
333 densable organic compounds are identifiable on the FTIR spectra in the first
334 events of thermal exposure. The main compounds released are formaldehyde,
335 methanol, formic acid, acetic acid and phenol compounds. The detection of these
336 gases is in agreement with the literature [29, 32] (Table 3), which generally ascribe
337 their formation to fragmentation and/or breaking of chemical groups of wood
338 polymers. Besides, the oxygen level does not alter the detection of these organic
339 compounds nor the peak of release of formaldehyde in the early stage of the test
340 (Figure 6c). It suggests again that the first events in wood decomposition are
341 mainly depending on the heat level.

342 Then, as it blends with oxygen, this hot gaseous production oxidizes and igni-
343 tion occurs. Thus, the previous mentioned organics compounds are no more
344 detected on FTIR spectra while the production of CO₂ greatly increases. At igni-
345 tion, the CO₂ emission peaks around 30000 ppm for the three oxygen levels and
346 then reaches a steady state (Figure 6a). The high quantity of CO₂ released at igni-
347 tion is due to a rapid depolymerization of wood units that gives unstable interme-
348 diaries. These intermediaries contain carboxyl and carbonyl groups that undergo
349 fragmentation [32], for example:



351



353 A small peak of CO, due to this depolymerization, is observed at ignition too
355 (Figure 6b) for all flaming specimens. However, at 15 vol% O₂ and from 750 s,
356 the CO production increases to reach a steady state and the organic species
357 observed at ignition as well as ethene are detected. Then, from 1700s and at both
358 18 and 15 vol% O₂, a peak in CO emission occurs. This increase may be related
359 to the lack of oxygen shifting the reaction of combustion towards incompleteness.
360 Indeed, as discussed previously, flames are less intense and located on the edges of
361 the specimen. The CO/CO₂ ratio is greater than 0.05, which means that the reac-

Table 3
Organic Compounds Detected in FTIR Analysis and Their Possible Way of Formation

Organic compound	Possible way of formation [32]
Formaldehyde (CH ₂ O)	Fragmentation of C–C bond linked to hydroxyl groups (lignin)
Methanol (CH ₃ OH)	Fragmentation of methoxy groups (hemicellulose and lignin)
Formic acid (HCOOH)	Rupture of carboxylic acid functions (hemicellulose)
Acetic acid (H ₃ CCOOH)	Fragmentation of acetic substituents (hemicellulose) Reaction of C–C bonds within and between alkyl chains (lignin)
Phenol compounds	Breaking of glycosidic linkages (cellulose)
	Breaking of ether bonds between monomers units (lignin)
	Reaction of C–C bonds within and between alkyl chains (lignin)

tion of combustion is incomplete. An increase in CH₄ (Figure 6e), which is released from demethylation reactions of the remaining methyl (–CH₃) substituents of the residue, occurs at the same time and thus argue in favor of the combustion reaction being below stoichiometry. The more methane is produced, the more aromatic the char is [32].

For the non-flaming specimen (10 vol% O₂), in the first events of thermal exposure, the same condensable organic compounds as flaming specimens are identifiable on the FTIR spectra (Table 3). However, as the low level of oxygen slows down the kinetics of decomposition and limit oxidation, additional gases such as acetaldehyde (C₂H₄O), methane and ethene (C₂H₄) are detected [25]. As a result, a high and broad peak of formaldehyde is produced at the beginning as well as one of acetaldehyde (Figure 6c and d). Acetaldehyde can be released from the thermal decomposition of levoglucosan (C₆H₁₀O₅), which is an intermediate compound released from cellulose decomposition [29]. Then, a strong and broad peak of CO production follows it while the CO₂ production is very low, with a CO/CO₂ ratio of 1.37. Indeed, there is not enough oxygen to oxidize the CO produced into CO₂. Thus, the production of unburnt hydrocarbon species is also promoted, with a high production of methane throughout the experiment (Figure 6e) while a peak of ethene (23 ppm) around 120 s is detected and is followed by a peak of propene (15 ppm).

For flaming specimens (21, 18 and 15 vol% O₂), oxygen decrease favors incomplete combustion at the end of the test with an increase in CO and CH₄ production. For non-flaming conditions, smoldering promotes the production of aldehydes (formaldehyde and acetaldehyde) and unburnt gases. Thus, the production of dangerous gases is favored at low oxygen level and when the combustion is non-flaming.

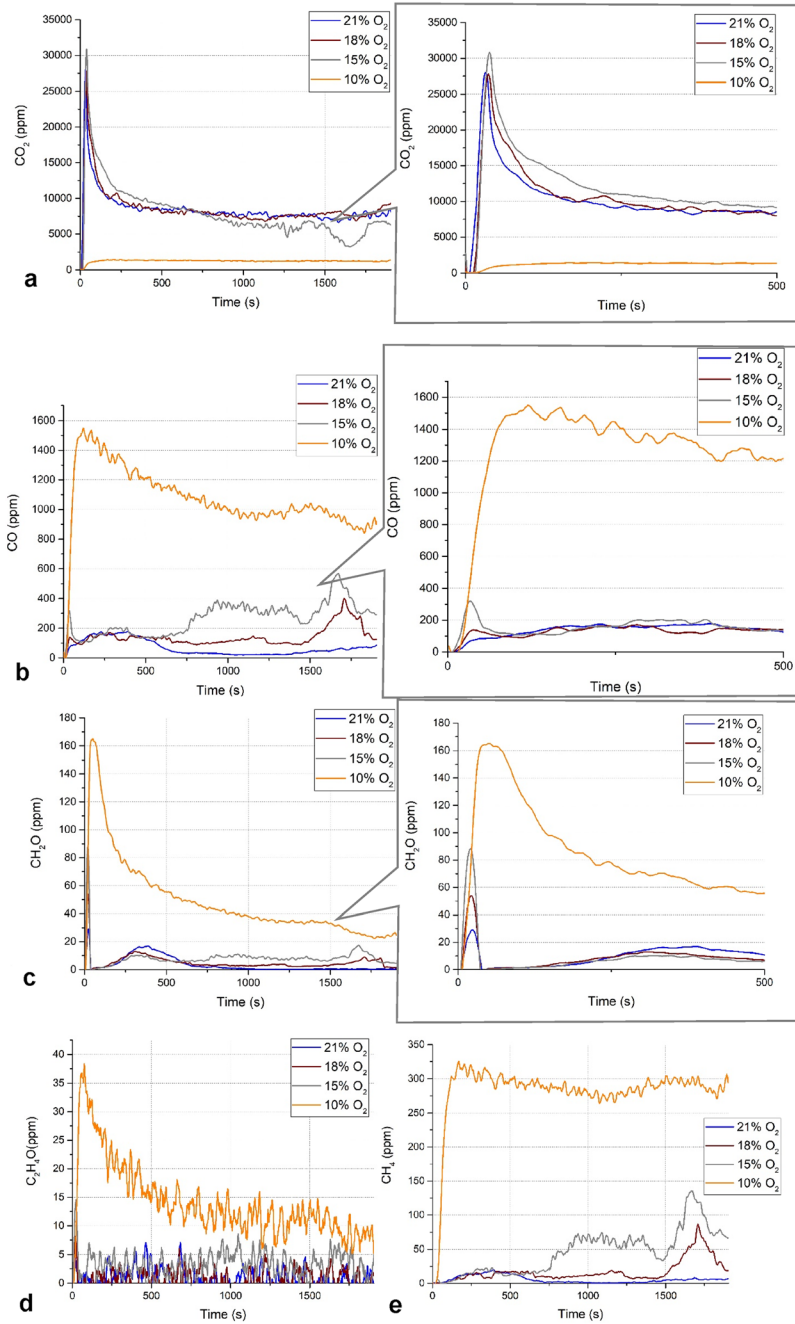
Effects of Oxygen Concentration on the Reaction to Fire of Cross-Laminated Timber

Figure 6. Influence of the oxygen level the gaseous emissions at 50 kW/m²: (a) CO₂, (b) CO, (c) CH₂O, (d) C₂H₄O and (e) CH₄.

388 3.2. Aerosol Production

389 To complete the characterization of the gas phase, the influence of oxygen on the
390 aerosol size distribution as well as the concentration of aerosol is now investi-
391 gated.

392 At ignition, for the three burning samples (21, 18 and 15 vol% O₂), the
393 distribution size is bimodal and centered on the modes 0.267 m and 0.109 m
394 (Figure 7a). This corresponds to the accumulation mode of aerosols (0.1–1 m)
395 which indicates the agglomeration and growth of particles [33]. The concentration
396 of aerosols in these modes is the highest at 15 vol% O₂ but the orders of magni-
397 tude are close. Then, the bimodal distribution is crushed by a great increase in the
398 mode 0.03 m (Figure 7b). Consequently, the distribution becomes monomodal
399 and centered on the mode 0.03 m. This now corresponds to a nucleation mode
400 (diameter inferior to 0.1 m) representing the formation of fresh aerosols. This may
401 be due to the nucleation of volatiles particles as the effluent mixture cools down
402 after ignition. Indeed, vaporized gases condensed and became particles having
403 very small size [33], [36, 37]. Globally, the concentration in this mode is of the
404 same magnitude at 21, 18 and 15 vol% O₂.

405 For 10 vol% O₂, the combustion is non-flaming and the distribution is mono-
406 modal centered on the mode 0.03 m (Figure 7a). Compared to the flaming speci-
407 mens, the concentration in the mode 0.03 m at 10 vol% O₂ is slightly higher
408 (Figure 7b). In some studies, the lack of oxygen is shown to increase the number
409 of particles. Indeed, with less oxygen available, the number of unburnt particles
410 increases [36, 37].

411 For the flaming specimens, a bimodal distribution centered on the modes
412 0.267 m and 0.109 m is observed at ignition. Then, it is replaced by a monomodal
413 distribution centered on the mode 0.03 m. For the non-flaming specimen, the dis-
414 tribution is monomodal centered on the mode 0.03 m. Globally, the distribution
415 in size of aerosols is not influenced by oxygen level but by the combustion pro-
416 cess.

417 The influence of oxygen on the reaction to fire, the gas and the aerosol produc-
418 tion of CLT was fully assessed at 50 kW/m². To verify if similar behaviors are
419 observed, it is crucial to study the influence of oxygen on less aggressive fire sce-
420 narios.

421 3.3. Influence of Oxygen Level at 20 kW/m²

422 The reaction to fire of CLT is also strongly dependent on the thermal attack, thus
423 the influence of the heat flux was evaluated at 20 kW/m². This thermal attack
424 enables to study less aggressive fire scenarios, such as smoldering, with slower
425 decomposition kinetics.

426 3.3.1. Mass Loss and Heat Release 3.3.1.1. Mass Loss The evolutions of remain-
427 ing mass as a function of time (Figure 9a) are very slow and similar at the begin-
428 ning of the thermal exposure whatever the oxygen concentration. However,
429 around 550 s for 21 vol% O₂, and around 1042 s for 18 vol% O₂, the samples
430 ignite and the mass fraction of CLT drops while the MLR reaches a peak (Fig-

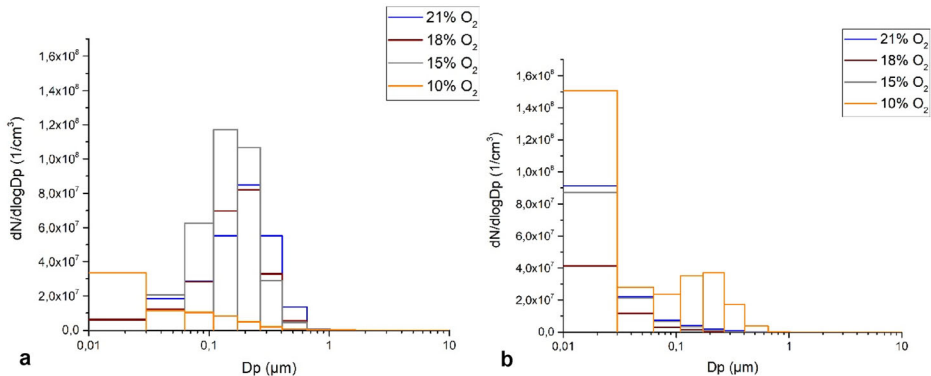
Effects of Oxygen Concentration on the Reaction to Fire of Cross-Laminated Timber

Figure 7. Distribution size of aerosols at 50 kW/m² at: (a) 1484 s.

431 ure 9b). The time to reach the peak of MLR more than doubles between 21 and
 432 18 vol% O₂ and the pMLR is the highest for 21 vol% O₂ (Table 4). The lack of
 433 oxygen delays the ignition [38, 39] as well as the flame's temperature [40], which
 434 reduces the solid degradation rate. In the end, the samples flame out to reach a
 435 steady state around 0.25 g/s. Furthermore, the amount of CLT residue after
 436 32 min is 68% at 21 vol% O₂ whereas it is 77% at 18 vol% O₂. The pyrolysis
 437 front has reached higher thickness at 21 vol% O₂ than at 18 vol% O₂ as the char
 438 formed is less protective (Figure 8).

439 At 15 and 10 of oxygen, the decomposition occurs without flames. The mass
 440 losses (Figure 9a) are similar until 700 s where the decomposition rate at 15% O₂
 441 slightly increases. As a result, the MLRs follow a slow increase in the beginning,
 442 as water evaporates, and tend to be constant for the rest of the test (Figure 9b).
 443 The amount of CLT residue after 32 min is 81% at 15 vol% O₂ and 82% at 10
 444 vol% O₂ (Figure 8c and d). At these low concentrations and under 20 kW/m², the
 445 influence of oxygen does not seem to have a significant impact on the decomposi-
 446 tion of the CLT.

447 In the condensed phase, as at 50 kW/m², the oxygen concentration affects the
 448 combustion process (flaming or non-flaming) and the kinetics of decomposition.
 449 The higher the oxygen content is the faster the mass loss is.

450 **3.3.1.2. Ignition and Heat Release** In the gas phase, the HRR curves depict two
 451 combustion behaviors (Figure 10). Indeed, at 21% and 18% of oxygen, a peak of
 452 heat release from flaming combustion is observed while at 15% and 10% of oxy-
 453 gen the non-flaming decomposition is depicted by a slow and low increase in
 454 HRR. For flaming samples, the peak of HRR is reduced of 48% between the
 455 peak of HRR at 21% O₂ and the peak of HRR at 18% O₂. This is due to the low
 456 oxygen amount that does not allow a complete reaction neither a high tempera-
 457 ture of flame. After ignition, both samples rapidly flame out. For lower oxygen
 458 levels, the HRR increases slowly to reach a steady state around 20 kW/m². As the
 459 behaviors are similar, the influence of oxygen seems again negligible.

Table 4
Influence of Oxygen on the Fire Parameters at 20 kW/m²

Oxygen level (%)	t _i (s)	pMLR (g/s)	pHRR (kW/m ²)
21	400	0.061	96
Standard error	105	0.003	4
18	1042	0.041	51
Standard error	257	0.021	29
15	–	0.023	20
Standard error	–	0.034	4
10	–	0.024	22
Standard error	–	0.022	13

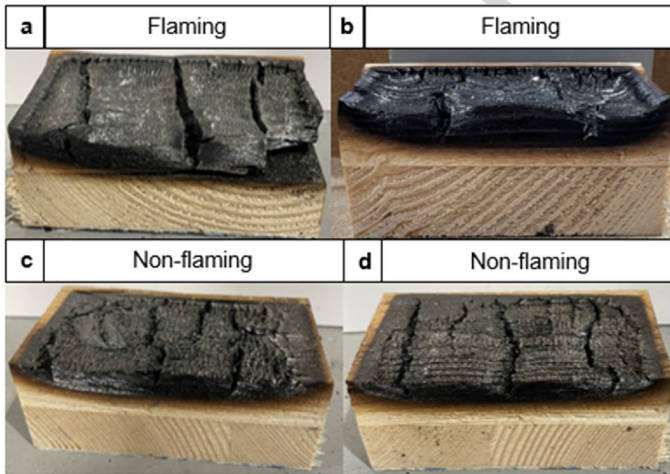


Figure 8. Sliced CLT after 32 min of test at 20 kW/m² at (a) 21% O₂, (b) 18% O₂, (c) 15% O₂ and (d) 10% O₂.

460 In summary, at 20 kW/m², two behaviors are observed according to the oxygen
461 concentration: flaming for 21% and 18% O₂ and non-flaming for 15% and 10%
462 O₂. The mass loss and heat release rates are similar whatever the oxygen level
463 O₂. The mass loss and heat release rates are similar whatever the oxygen level
464 when the combustion is non-flaming. For flaming combustion, the ignition is
465 delayed and less intense with oxygen decrease.

465 3.3.2. Gas Production For the flaming specimens (21 vol% O₂ and 18 vol% O₂),
466 in the first events of thermal decomposition, CLT released ethene and the same
467 organic compounds as it did at 50 kW/m², namely formaldehyde, methanol, for-
468 mic acid, acetic acid and phenol compounds (Table 3). At 21 vol% O₂, before
469 200 s, a peak of formaldehyde occurs (Figure 11c). Then, at 21 vol% O₂ and from
470 300 s, another peak of formaldehyde is reached as well as one of CO and CH₄
471 (Figure 11b and d). At 400 s, the sample ignites and a peak in CO₂ occurs (Fig-

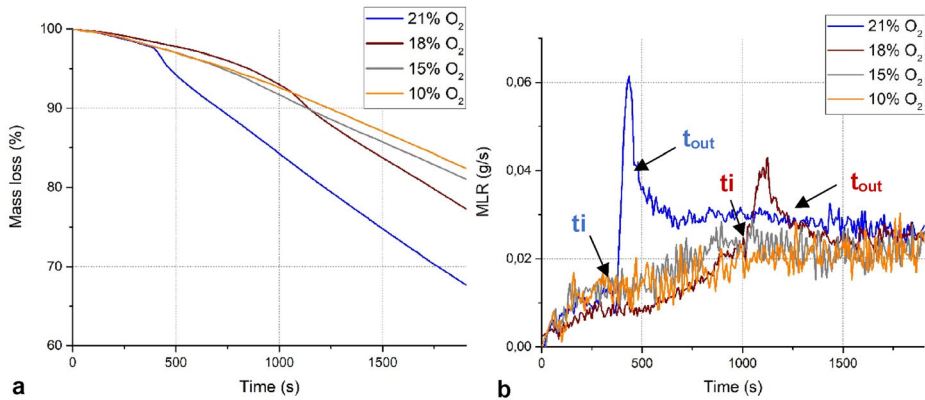
Effects of Oxygen Concentration on the Reaction to Fire of Cross-Laminated Timber

Figure 9. Effect of oxygen concentration on: (a) the mass loss, (b) the MLR.

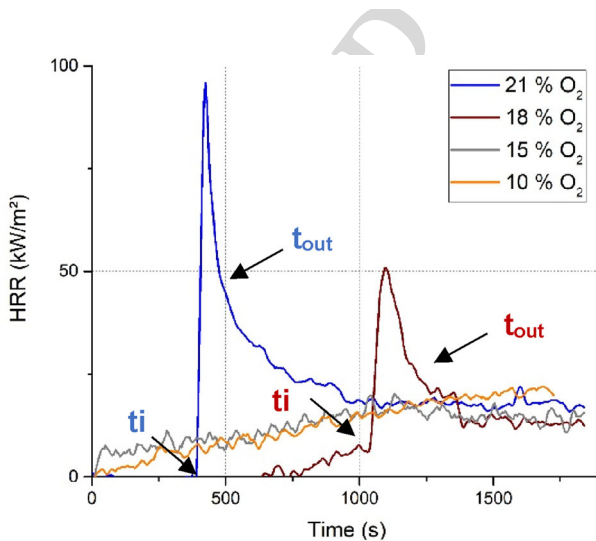


Figure 10. Effect of oxygen concentration on the HRR.

472 ure 11a). The CO₂ concentration rapidly decreases until the sample flames out. As
 473 a result, peaks of CH₂O, CO and CH₄ arise at 1050 s. The behavior is similar at
 474 18 vol% O₂ but it is delayed in time. Indeed, the second peak of formaldehyde
 475 (Figure 11c) as well as these of CO and CH₄ are reached at 1035 s (Figure 11b
 476 and d). CLT ignites at 1042 s with a peak of CO₂ emission and then flames out at
 477 1160 s with an increase in CH₂O, CO and CH₄. At the end of the test, the pro-
 478 duction of gases at 18 vol% O₂ reaches a steady state instead of decreasing as at
 479 21 vol% O₂. In general, for both conditions, the CO/CO₂ is greater than 0.05 dur-
 480 ing the whole test (incomplete combustion). Moreover, the production of carbon
 481 dioxide is slightly higher at 21 vol% O₂ than at 18 vol% O₂ while it is the oppo-
 482 site for carbon monoxide and methane production.

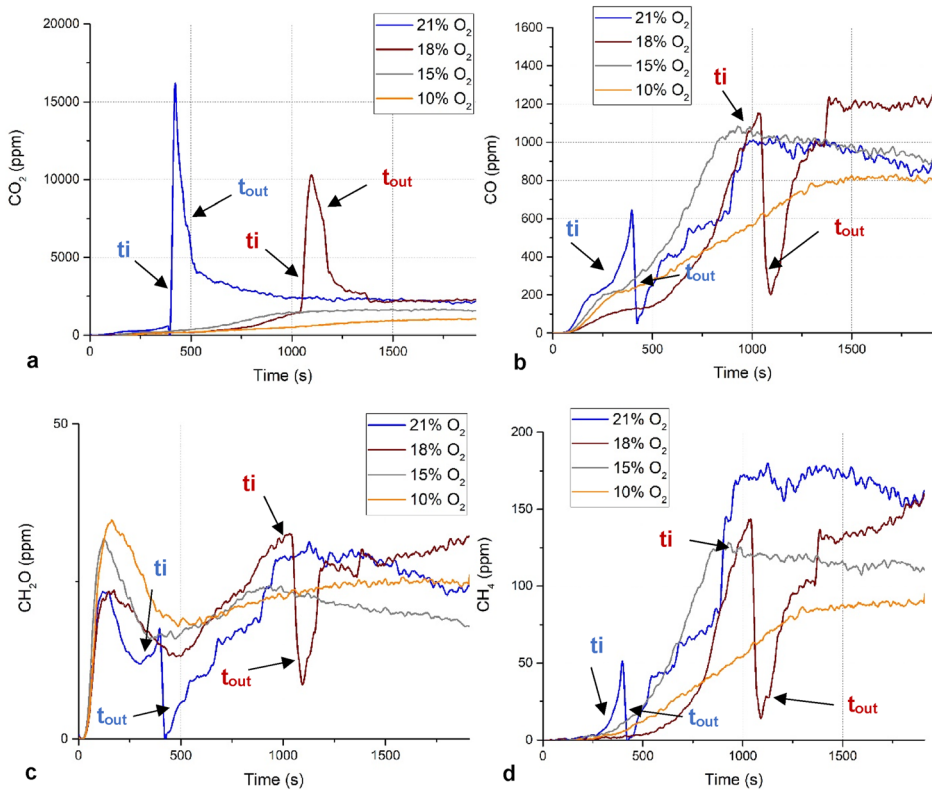


Figure 11. Influence of the oxygen level the gaseous emissions at 20 kW/m²: (a) CO₂, (b) CO, (c) CH₂O and (d) CH₄.

483 For non-flaming combustion (15% and 10% O₂), FTIR spectra reveal that the
 484 organic compounds produced are the same as for the flaming specimens and that
 485 they are produced during the whole test. For both oxygen levels, the CO/CO₂
 486 ratio is greater than 0.05 during the whole test as the production of CO₂ is very
 487 low (Figure 11a). At the beginning of the test, before 250 s, a peak of formalde-
 488 hyde comes up (Figure 11c) as wood decomposes and then the production drops
 489 down. In the meantime, the production in CO and CH₄ significantly increases, as
 490 wood is carbonizing, and they reach a maximum at 935 s at 15 vol% O₂ and
 491 1500 s at 10 vol% O₂. It slightly decreases after (Figure 11a and d) and an
 492 increase in formaldehyde is also observed at these times. The same behaviors are
 493 observed at 15 vol% O₂ and 10 vol% O₂, but again the phenomena are delayed in
 494 time with oxygen decrease. For non-flaming conditions, the production of CO and
 495 CH₄ is promoted with the increase of oxygen concentration.

496 For flaming specimens, oxygen decrease favors incomplete combustion with
 497 higher production of CO and CH₄. However, for non-flaming conditions, the
 498 higher the oxygen level is the higher the production of CO and CH₄ is.

499 3.3.3. *Aerosol Production* At ignition, for the burning samples at 21% O₂ and
500 18% O₂, the distribution is bimodal and centered on the modes 0.267 m and
501 0.109 m (Figure 12a), which corresponds to the accumulation mode. Then, as for
502 50 kW/m², the bimodal distribution is crushed by a great increase in the mode
503 0.03 m. Consequently, the distribution becomes monomodal and centered on the
504 mode 0.03 m (Figure 12b). This corresponds again to the nucleation mode. The
505 concentration in aerosols is slightly higher at 21% O₂ but globally of the same
506 magnitude.

507 For the non-flaming specimens, the distribution is also monomodal centered on
508 the mode 0.03 m during the whole test (Figure 12a and b). The concentration in
509 the mode 0.03 m is similar between 15 and 10% O₂. However, it is slightly
510 reduced compared to 21 and 18% O₂ samples. It can be due to the change in the
511 combustion process, here smoldering which induces slower decomposition rates
512 [34].

513 For the flaming specimen, a bimodal distribution centered on the modes
514 0.267 m and 0.109 m is observed at ignition at 21% O₂ and at 18%. Then, it is
515 replaced by a monomodal distribution centered on 0.03 m. For the non-flaming
516 specimen, the distribution is monomodal centered on the mode 0.03 m.

517 3.4. *General Discussion*

518 The fire behavior as well as the gas and aerosol production of CLT according to
519 the oxygen concentration and the heat flux was investigated. These latter param-
520 eters are limiting factors to enable combustion as well as altering the kinetics of
521 decomposition. The combination of both of them highlights different fire scenarios
522 where the quantity of gases produced can be potentially toxic.

523 The first type of decomposition scenario obtained with CLT is flaming combus-
524 tion (Figure 15). At 50 kW/m², the oxygen level is not high enough to disturb the
525 combustion regime in the early stage of the test and the condensed phase is not
526 affected. Indeed, the times to ignition (Table 2) and the maxima of MLR are simi-
527 lar at 21, 18 and 15 vol% O₂ (Figure 13). It could be assumed that the surface
528 oxidation reactions would contribute to the thermal decomposition of CLT but Di
529 Blasi [41] claims that the flame prevents the char oxidation. Oxygen cannot diffuse
530 through the flame as it is consumed at its boundaries. Moreover, according to
531 Kashiwagi [42], the region near the flame is poor in oxygen because of the
532 homogenous reactions of combustion. As a result, all oxidative reactions near the
533 surface stop. These observations are in accordance with the fact that oxygen diffu-
534 sion to the surface of CLT is limited by the increased mass heat flux of gaseous
535 products over time. As a result, the heterogeneous reactions of the solid phase
536 cannot occur once the flame is established. However, the oxygen concentration
537 does affect the gas phase. With oxygen decrease, the flaming combustion shifts in
538 time to incompleteness with higher CO and CH₄ yields (Figure 14). The oxidation
539 reactions of organic compounds into CO₂ are disadvantaged against oxidation
540 into CO as well as demethylation at low oxygen levels. It should be noted that
541 although the combustion regime is slightly altered with oxygen decrease, the yields
542 in CO₂ are marginally affected. Furthermore, lowering the heat flux to 20 kW/m²

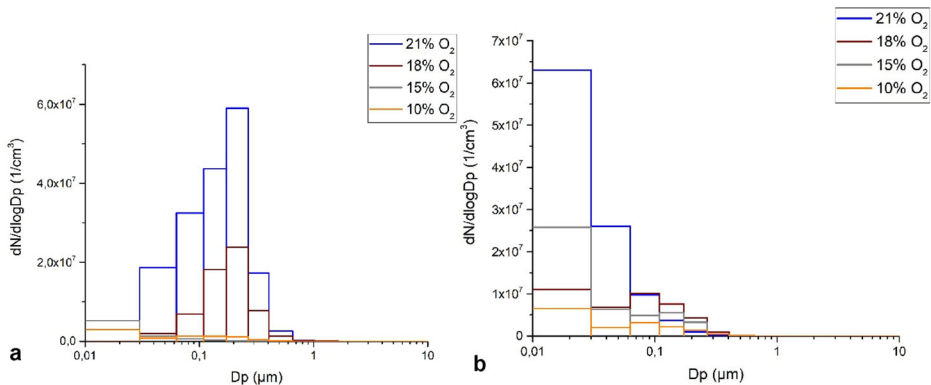


Figure 12. Distribution size of aerosols at 20 kW/m² at: (a) t_i , (b) 1484 s.

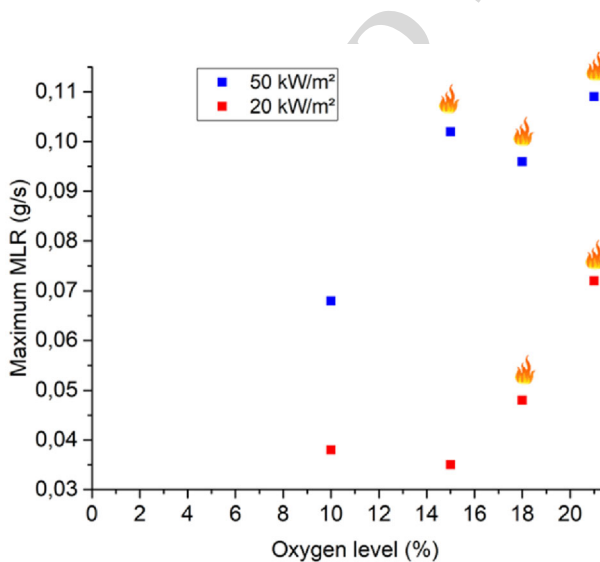


Figure 13. Effect of oxygen and heat flux on the maximum MLR.

543 leads to slower kinetics of decomposition (20 kW/m², 21 vol% O₂ and 20 kW/m²,
 544 18 vol% O₂). As less heat is received at the surface of CLT, the ignition process is
 545 both governed by heat and oxygen concentration. At 21 vol% O₂, the ignition of
 546 CLT is 22 times delayed in time compared to 50 kW/m². Similarly, at 20 kW/m²,
 547 the time to ignition at 18 vol% O₂ is increased by a factor of 2.5 compared to the
 548 time to ignition at 18 vol% O₂. Moreover, the pMLRs are greatly lowered compared
 549 to 50 kW/m² (Figure 13). Between 50 and 20 kW/m², the pMLRs are
 550 reduced by 34% at 21 vol% O₂ and of 50% at 18 vol% O₂. Consequently, the
 551 wood matrix has time to rearrange into a more stable matrix. Indeed, before a
 552 bond can be completely broken from the wood matrix, new bonds could form to

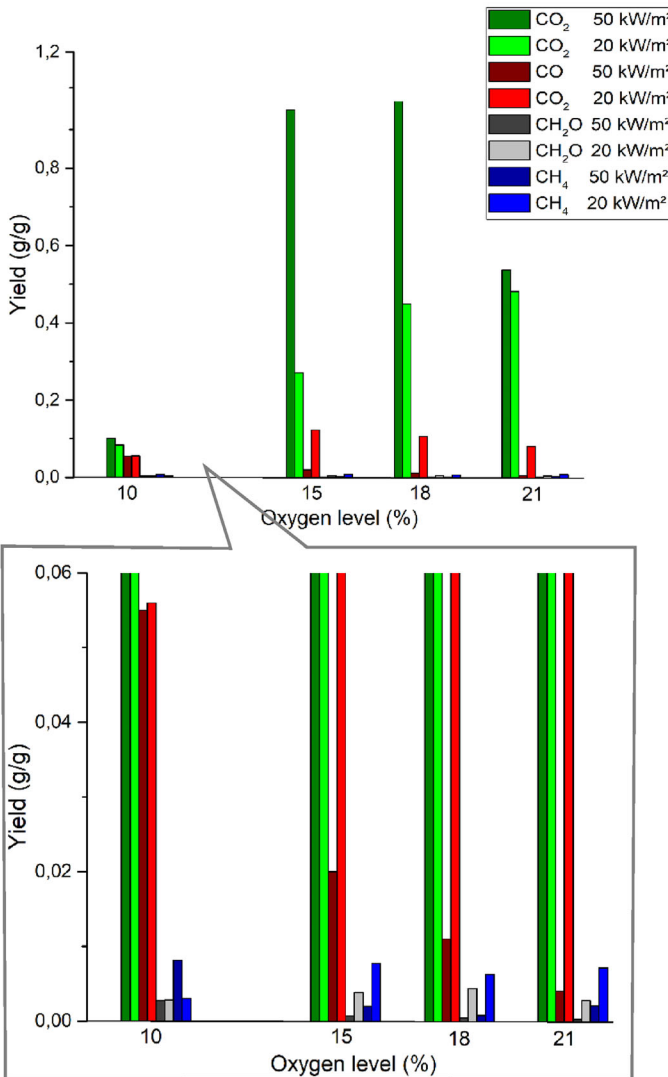
Effects of Oxygen Concentration on the Reaction to Fire of Cross-Laminated Timber

Figure 14. Total yields of CO₂, CO, CH₂O and CH₄ as a function of oxygen level and heat flux.

553 bond the fragment back to the previous wood matrix [43]. Thus, less organic vola-
 554 tiles are produced while the yields of CO and CH₄ are promoted (Figure 14). As
 555 CLT rapidly flames out, a dramatic increase in CO and CH₄ occurs, which is
 556 amplified with oxygen decrease. For all types of flaming scenarios described, the
 557 distribution of aerosol particle size is centered on the modes 0.267 and 0.109 m at
 558 ignition and then it shifts to the mode 0.03 m. Increasing the heat flux increases
 559 the number of particles per cm³, which is a risk as submicronic particles can pene-
 560 trate the lungs [37].

561 The second type of decomposition scenario of CLT is non-flaming combustion
562 (Figure 15). The oxygen vitiation affects the combustion process. Indeed, at
563 50 kW/m^2 , though the MLR curve at 10 vol% fits the shape of the flaming speci-
564 men (21, 18 and 15 vol% O_2) (Figure 3a), there is not enough oxygen to ignite the
565 released volatiles and only smoldering occurs. However, it should be highlighted
566 that below 21 vol% O_2 at 50 kW/m^2 , the MLR curves of flaming and non-flaming
567 specimens are close in value (around 0.4 g/s) during the steady state reached after
568 the peak. This again suggests that the heat flux mainly governs the decomposition
569 process and that oxygen has a low impact on it. It is also interesting to note that
570 the pMLR reached at 10 vol% O_2 is similar to that at 21 vol% O_2 at 20 kW/m^2
571 (at ignition) (Figure 13). It confirms the limiting role of oxygen in the combustion
572 regime. Moreover, the lack of oxygen increases the production of $\text{C}_2\text{H}_4\text{O}$ (Fig-
573 ure 6d) and the yields of CH_4 and CO as there is not enough oxygen to oxidize
574 organic volatiles into CO_2 (Figure 14). At high heat flux, non-flaming combustion
575 is a threat to fire safety as the production of unburnt gases is promoted and the
576 decomposition and heat release rates remain high. Moreover, the production of
577 aerosols is significant which is all the more dangerous as the distribution size of
578 aerosols is centered on the mode 0.03 m (lung penetration). Reducing the heat
579 flux to 20 kW/m^2 leads to a higher demand in oxygen to have a flaming combus-
580 tion. Indeed, the combustion is not flaming at 15 vol% O_2 whereas it was flaming
581 at 50 kW/m^2 . The low heat received at the CLT surface also results in lower
582 decomposition rates as the pMLR is reduced by 63% between the 10 vol% O_2
583 cases (Figure 13). It should be underlined that the pMLR at 15 and 10 vol% O_2
584 are similar (Figure 13). Thus, the oxygen has no significant impact on the decom-
585 position of the condensed phase. Moreover, the yields of CO_2 and CO are similar
586 at 10 vol% O_2 between 50 and 20 kW/m^2 while those of CH_4 are slightly higher
587 at 50 kW/m^2 (Figure 14). This highlights the low impact of the heat flux on gas
588 production when the combustion is not flaming. Finally, for non-flaming combus-
589 tions, the distribution of particle size is also centered on the mode 0.03 m but with
590 a lower concentration than for flaming conditions.

591 **4. Conclusion**

592 The assessment of the fire behavior, the gas production and the aerosol size distri-
593 bution of Cross-Laminated Timber were carried out with a homemade (based on
594 the ISO 5660-5 standard) Controlled-Atmosphere Cone Calorimeter coupled with
595 a FTIR and an ELPI. This is an appropriate approach to examine the effect of
596 the oxygen and the irradiance level on the solid/gas phase phenomena occurring
597 during the thermal degradation of CLT. Both heat flux and oxygen are revealed
598 to be limiting factors in the combustion process, which enables the classification
599 of fire behaviors of CLT (Figure 16).

600 In general, oxygen vitiation shifted the reaction of combustion towards incom-
601 pleteness or even prevented it in some cases. It also greatly promoted the emission
602 of unburnt species such as carbon monoxide and methane. These phenomena are
603 favored at reduced heat flux. For all flaming specimens, the aerosol size distribu-

Effects of Oxygen Concentration on the Reaction to Fire of Cross-Laminated Timber

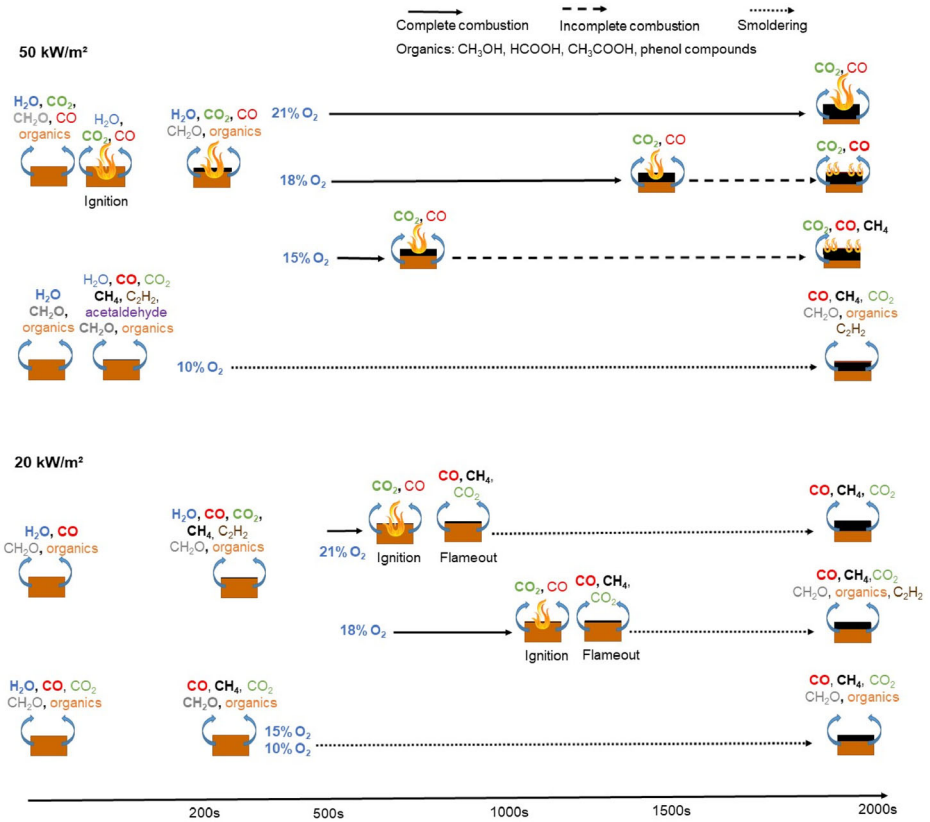


Figure 15. Flaming and non-flaming decomposition of CLT.

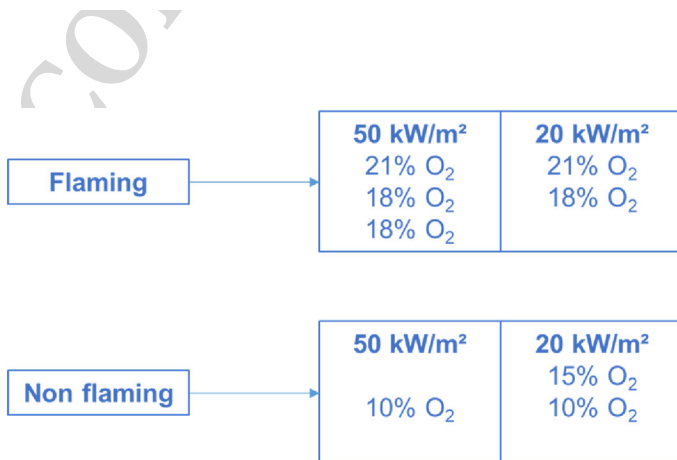


Figure 16. Classification of fire behavior of CLT according to oxygen level and heat flux.

tion is centered on the modes 0.267 m and 0.109 m at ignition and then shifts to the mode 0.03 m, while for non-flaming specimens the distribution size of aerosols is centered on the mode 0.03 m.

This study highlights the hazards of different fire scenarios. CLT exposed to a high heat flux combined with low oxygen level creates an environment with a significant amount of dangerous gases and submicronic aerosols. Besides, smoldering is a hazard for fire spread in a room. Further investigations on the lower flammability limit of CLT according to oxygen level and heat flux would be interesting to have a finer understanding of its behavior. A finer measuring range of the distribution size of aerosols could be also beneficial to better assess the aerosol production.

Funding

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