Construction and Building Materials 317 (2022) 125931

Contents lists available at ScienceDirect

Construction and Building Materials

journal homepage: www.elsevier.com/locate/conbuildmat

Lightweight cement composites containing end-of-life treated wood – Leaching, hydration and mechanical tests

Sara Dias [a], João Almeida [a,b,*], Bruna Santos [a,c], Pedro Humbert [a,c], António Tadeu [a,c], Julieta António [a,c], Jorge de Brito [d], Pedro Pinhão [e]

[a] Itecons - Institute for Research and Technological Development in Construction, Energy, Environment and Sustainability, Rua Pedro Hispano s/ n., 3030-289 Coimbra, Portugal
[b] Chemistry Centre, Department of Chemistry, University of Coimbra, Rua Larga, 3004-535 Coimbra, Portugal
[c] ADAI, LAETA, Department of Civil Engineering, University of Coimbra, Pólo II, Rua Luís Reis Santos, 3030-788 Coimbra, Portugal
[d] CERIS-ICIST, Instituto Superior Técnico, University of Lisbon, Av. Rovisco Pais 1, Lisbon 1049-001, Portugal
[e] Toscca - Equipamentos em Madeira, Lda. Zona Industrial Oliveira de Frades, Lote 71/72, 3680-171 Oliveira de Frades, Portugal

ARTICLE INFO

Keywords:
Wood waste
Release of dangerous substances
Wood-cement compatibility
Hydration heat
Physical-mechanical properties
Circular economy

ABSTRACT

A huge amount of treated wood waste containing hazardous substances is produced every year all over Europe that simply ends up in landfills. In this study, the authors investigate the practical feasibility of using end-of-life treated wood for producing innovative lightweight cement composites. For this purpose, wood waste from four different sources was characterised to assess the presence of leaching substances and to ascertain physical properties relevant to the production of cement composites. Afterwards, cement pastes containing, firstly, wood extractives, and later, wood particles were prepared to assess the compatibility between the wood and Portland cement binder. To improve the wood-cement compatibility, additional tests were carried out using extracted and chemically treated wood particles. The results were discussed in terms of hydration heat and mechanical properties. Finally, a concrete mix containing selected wood particles was produced for a preliminary examination of the effect of wood waste on the mechanical properties of a reference concrete.

The results show that the concentration of leaching substances can vary significantly depending on the source of wood waste. Small variations were found in the hydration heat profile of different cement composites containing up to 10 %(w/w) of wood particles, contrasting with the significant reduction found in compressive strength. The results of the concrete mixes further show that the incorporation of wood particles significantly reduces the mechanical performance of the hardened material, though such composites can still be used in applications with low-structural requirements.

1. Introduction

According to EUROSTAT [1], the EU28 countries generated every year more than 48 million tonnes of wood waste from multiple sources (e.g. construction, agriculture and railway). It turns out that a significant part of this waste is contaminated with hazardous substances used to protect wood from biological degradation and ageing. The seriousness of the ecological risk and the human hazard of wood preservatives have been highlighted in several studies in the last few years [2–6].

Although treated wood residues are mainly composed of cellulose, hemicellulose, and lignin, small amounts of several heavy metals and organic compounds can also be found in their composition, which limits the recycling options [7,8]. Disposal in landfills remains thus the commonest way to deal with end-of-life treated wood. However, increasing awareness about the scarcity of resources and environmental concerns with the disposal of waste in landfill have motivated researchers to investigate innovative recycling strategies.

Several recycling approaches can be exploited to add value to end-of-life treated wood, namely the production of wood-based composites. However, many industries are reluctant to incorporate wood fibres containing chemical preservatives in new materials due to health and environmental concerns, and they most often opt to use non-treated

* Corresponding author at: Itecons - Institute for Research and Technological Development in Construction, Energy, Environment and Sustainability, Rua Pedro Hispano s/ n., 3030-289 Coimbra, Portugal.
E-mail address: joao.almeida@itecons.uc.pt (J. Almeida).

https://doi.org/10.1016/j.conbuildmat.2021.125931
Received 30 September 2021; Received in revised form 21 November 2021; Accepted 26 November 2021
Available online 8 December 2021

wood. Mohajerani et al [8] claim that wood-based composites are not an ideal solution as they are simply deferring disposal rather than avoiding it. However, the same authors recognise that the reuse of treated wood in construction applications is a reasonable solution if the results of leaching tests (e.g. [9,10]) comply with regulatory limits. Following this path, several studies have investigated the environmental risk of reusing treated wood [11–14]. For instance, Qi [14] performed leaching tests on wood particles of CCA-treated red pine and wood-cement boards consisting of ordinary Portland cement and the same wood particles, concluding that the copper and arsenic were fixed quite well in the board, as opposed to the situation with the wood particles.

Note, however, that the environmental and health risks are not the only topic of concern when incorporating wood particles in cement composites. Major compatibility issues have been reported between some wood species and cement binders. Wood extractives, in particular, have been shown to have an important role in disturbing the chemical processes of cement hardening [15]. However, the impact of this phenomenon on the mechanical performance of wood-cement composites is difficult to predict, as it depends not only on the extractive content, but also on the chemical composition of the extractives. Moreover, other physical characteristics of the wood particles (e.g. size and geometry distribution, surface area and water absorption capacity) and composition characteristics of the mix (e.g. cement type, wood-cement ratio and water-cement ratio) contribute to the final behaviour of the composite.

A very useful method to assess the chemical compatibility between wood and cement is to compare the heat release during the exothermic process of cement hydration of a wood-cement mix with that of a corresponding cement paste without any wood. Information on the hydration rate during the initial phase of cement hardening can also be used to complement this assessment. For this purpose, several test standards can be followed (e.g. EN 196–11:2018, ASTM C1702-17). Fan et al. [16], for example, investigated the compatibility of ordinary Portland cement with 15 commercially available wood species by studying cement hydration and concluded that the heat release of wood-cement pastes varies considerably, depending on the wood species. The same authors showed that wood-cement compatibility can be significantly improved by pre-treating wood particles with a saturated lime ($Ca(OH)_2$) solution. Miller and Moslemi [17] also studied nine wood species from North America and concluded that hardwoods have a stronger negative effect on the hydration of cement and tensile strength of composites than softwoods do. They also found that heartwood (central wood of trees) is more prone to affect the hydration of cement pastes than sapwood, which is probably because of the different solubility in water of saccharides present in the respective natural fibres. Kochova et al. [18] studied the effect of different organic compounds present in lignocellulose fibres on the cement hydration reactions, demonstrating the major role of glucose, mannose and xylose in cement hydration. These compounds slowed the hydration down for up to two days. Na et al. [19] also reported a greater delaying effect of glucose and sucrose compared to other compounds.

Regarding end-of-life treated wood, additional hazardous substances may be extracted depending on the preservatives used. Creosote is one of the oldest wood preservatives; it became widely used to protect timber elements that require a very long service life (e.g. bridgework, railway sleepers, marine pilings and utility poles). In addition to toxicity to fungi, insects, and marine borers, it serves as a natural water repellent. This tar-based preservative, which contains a complex mixture of natural oils, aromatic hydrocarbons (e.g. PAH and BTEX) and phenolic compounds, was extensively used before its carcinogenic properties became known. Although creosote is mostly insoluble in water, lower molecular weight compounds may become water-soluble after longer weather exposure and leach into the environment. The rest of the insoluble chemicals remain in a tar-like form until the end-of-life of the wood. Creosote is considered a restricted-use pesticide [20].

Chromated copper arsenate (CCA) was introduced later and quickly became one of the most common preservatives used to protect timber elements intended for outdoor applications. In the CCA treatment, the chromium acts as a fixing agent by helping the other chemicals to bind to the timber, also providing ultraviolet (UV) resistance. The copper protects the wood against decay, fungi, and bacteria, while the arsenic is the main insecticidal agent, protecting wood from insect-attacking, including termites and marine borers. The European Union restricts the use of arsenic, including CCA wood treatment [21]. Accordingly, CCA treated wood cannot be used in residential or domestic constructions, rather in industrial and public works (e.g. bridges, highway safety fencing, electric power lines and telecommunications poles).

Alkaline copper quaternary (ACQ) is a preservative made of copper and a quaternary ammonium compound (quat) like didecyldimethylammonium chloride. Copper is the primary bactericide and fungicide agent, while the quaternary ammonium cation is added to prevent growth of copper-tolerant bacteria, fungus, and mould, acting as an insecticide. ACQ has come into wide use following international restrictions on CCA. Its use is governed by international standards, which determine the volume of preservative uptake required for a specific timber end use (e.g. EN 351–1:2007, BS 8417:2011 + A1:2014). The main drawback of ACQ is the significant amount of ammonia released from treatment plants and freshly treated wood in storage yards.

However, these chemicals do not necessarily represent a problem when embedded in cement pastes [22,23]. Schmidt et al. [24] found stronger bendability of cement test pieces containing wood treated with chromated copper arsenate (CCA). The increased compatibility of CCA treated wood was attributed to the prior acid washing of water extractives from wood by CCA preservative. Huang and Cooper [25] also confirmed the good physical–mechanical properties of cement-bonded particleboards made from CCA-treated wood. More recently, the scientific community is getting more interested in exploring the potential of bio-based cementitious composites in construction by embedding non-conventional constituents in mortars and concrete [26–29]. Gloria et al. [26] proposed a method for designing mouldable bio-concretes, after studying cement composites containing three different vegetable residues (wood shaving, bamboo particles and rice husk). This procedure includes a physical and morphological characterization of the bio-aggregates, as well as the analysis of their compatibility with cement-based matrices. Other studies have also investigated the feasibility of using wood ashes as supplementary cementitious materials [30–32]. Such ashes are mainly composed of lime and silica, but are free of undesirable organic extracts. In addition, under given circumstances, they can show pozzolanic activity [33].

As previously mentioned, environmental concerns over the disposal of preservative-treated wood waste are the main driving forces of research into new recycling strategies. It is therefore necessary to look for innovative ways to treat wood waste. This work seeks to investigate the feasibility of using end-of-life treated wood for producing lightweight cement composites. For this purpose, wood waste samples from four different sources were characterized in terms of relevant chemical and physical properties. Cement pastes containing either wood extractives or wood particles were then used to assess the wood-cement compatibility based on hydration heat and mechanical properties. Further tests were carried out to screen the potential benefits of extracting or chemically treating wood particles before adding them to the cement paste. Finally, a concrete mix containing selected wood particles was produced for a preliminary examination of the effect of wood waste on the mechanical properties of a reference concrete.

Section 2 presents the materials and methods, fully describing all the chemical, physical, and mechanical tests. The results are presented in Section 3, with a comparison of hydration heat profiles and the mechanical performance of different wood-cement mixes. The main conclusions are summarised at the end (Section 4).

2. Materials and methods

Wood waste samples from various origins were first chemically and

physically characterised. Subsequently, to evaluate the wood-cement compatibility, two types of cement pastes were prepared: a Portland cement with wood waste extractives replacing the water; and a Portland cement with wood waste particles. Portland cement CEM II/B-L 32.5 N was used. Variations were introduced in terms of wood type, wood content, wood particle size, and wood pre-treatment. The analysis was carried out in two stages. First, 16 mixes were subjected to an isothermal calorimeter to evaluate the influence of the wood waste on the heat of hydration and the setting time of the cement pastes and wood-cement pastes. Then, the mixes with the best performance were subjected to mechanical tests. In both stages, a Portland cement paste was used as reference. Finally, a concrete mix containing selected wood particles were produced to initially examine the effect of wood waste on the mechanical properties of a reference concrete. All the materials and methods used in this work are detailed in following subsections.

2.1. Wood samples

Four types of wood waste were supplied in the form of chips (size up to 20 mm) by TOSCCA (a Portuguese firm), as illustrated in Fig. 1.

Information about the wood samples, including potential preservatives and estimated annual production, is summarised in Table 1. Note that the potential preservatives were identified based on the origin of the wood waste and decade of production.

2.1.1. Chemical characterisation

All the samples were first reduced to a particle size below 4 mm using a laboratory blade mill (Retsch, SM 100). Aqueous extracts of the different wood residues were then produced based on a one-stage batch test of 24 h at a liquid to solid ratio (L/S) of 10 L/kg dry matter according to EN 12457–2:2002. For this purpose, the sample material was brought into contact with an appropriate amount of deionized water in a polypropylene flask and mixed for 24 h at 10 rpm using a rotary shaker (VELP Scientifica, Rotax 6.8 overhead mixer). Note that this method, specifically developed to assess chemicals that can be leached from waste materials, assumes that equilibrium or near-equilibrium is achieved between the liquid and solid phases while the test is carried out. Subsequently, the solid residue was separated by filtration (0.45 μm pore filter) and the resulting eluates analysed by inductively coupled plasma optical emission spectrometry (Perkin Elmer, Optima 8000), gas chromatography associated with mass spectrometry (Bruker, SCION 456-GC, EVOQ TQ MS), liquid chromatography (Perkin Elmer, Flexar uHPLC), combustion-infrared (Analytik-Jena, multi N / C 3100) and other classic chemical methods. Given the nature and the amount of chemicals found in the eluate of WW4, the authors decided not to proceed with this wood waste in the production of cement pastes and the remaining tests.

Table 1
Information about the wood samples.

Sample reference	Wood source	Decade of production	Potential preservatives	Estimated annual production
WW1	Sawmills from untreated wood	2010–2020	—	1 500 m^3/year
WW2	Agriculture posts and fences	1990–2000	Alkaline copper quaternary (ACQ)	2 500 m^3/year
WW3	Agriculture posts and fences	1990–2000	Chromated copper arsenate (CCA)	10 000 m^3/year
WW4	Railway sleepers	1980–1990	Creosote	500 m^3/year

2.1.2. Physical characterisation

The previously mentioned wood samples, excluding WW4, were characterised in terms of water absorption, density and free surface water by adapting the procedures described in EN 1097–6:2013. Accordingly, the wood samples were dried at 105 °C for 24 h using a heating oven (Memmert, UF110) and ground into particles of varying size using a laboratory blade mill (Retsch, SM 100) coupled to different sieves. The three wood samples (WW1, WW2 and WW3) were characterised using particles $\leq$ 10 mm. The untreated wood sample (WW1) was additionally characterized using wood particles $\leq$ 4 mm, with a view to studying the influence of the particle size. A sample of 22 $\pm$ 4 g each of dried and ground wood was weighed and introduced into the pycnometer. To determine the progress of water absorption, the pycnometer was weighed after 5 min (m_0), 60 min (m_1), 120 min (m_2), 240 min (m_4), 330 min ($m_{4.5}$), 1260 min (m_{21}) and 1440 min (m_{24}). After the last weighing, the wood was left to drain for 5 min and weighed again. Then, the wood was gently dried with absorbent paper for 15 sec to remove the free surface water and weighed once again.

2.2. Wood-cement pastes and concrete mixtures

To proceed with the investigation of end-of-life wood-cement composites, several cement pastes containing wood extractives and wood particles were prepared varying the type of wood (WW1, WW2 and WW3), the wood particles content (5 w%, 10 w% and 15 w%), the particle size ($\leq$4 mm and $\leq$ 10 mm), and the pre-treatment of the wood (cold water extraction, hot water extraction, calcium hydroxide extraction and sodium silicate mineralization). A reference mix without wood was also prepared for comparison purposes. The experimental designs of the wood-cement pastes used in the hydration heat evaluation and mechanical tests are presented in Tables 2 and 3, respectively. For

Fig. 1. Wood waste samples used in this work.

Table 2
Wood-cement pastes (WCPs) used in the hydration heat evaluation (wood particle size = 10 mm).

Sample reference	Parameter	Wood waste type	Wood waste (w %)	Wood waste pre-treatment
Control	–	–	–	–
WECP_WW1_E24hCW	Extractives	WW1 extract (24 h cold water)	–	–
WECP_WW1_E48hCW		WW1 extract (24 h cold water)	–	24 h cold water
WECP_WW1_E2hHW		WW1 extract (2 h hot water)	–	–
WCP_5%WW1*	Wood type	WW1	5	24 h cold water
WCP_5%WW2		WW2	5	
WCP_5%WW3		WW3	5	
WCP_5%WW1*	Wood content	WW1	5	24 h cold water
WCP_10%WW1			10	
WCP_15%WW1			15	
WCP_5%WW1_4hCW	Wood pre-treatment	WW1	5	4 h cold water
WCP_5%WW1_6hCW			5	6 h cold water
WCP_5%WW1_8hCW			5	8 h cold water
WCP_5%WW1*			5	24 h cold water
WCP_5%WW1_2hHW			5	2 h hot water
WCP_5%WW1_24hCH			5	24 h Calcium hydroxide
WCP_5% WW1_24hSS1%			5	24 h Sodium silicate 1%
WCP_5% WW1_24hSS10%			5	24 h Sodium silicate 10%

*Same sample

Table 3
Wood-cement pastes (WCPs) used in the mechanical characterisation (wood particles were immersed in water for 24 h before inclusion in the cement paste).

Sample reference	Parameter	Wood waste type	Wood waste(w %)	Particle size (mm)
Control	–	–	–	–
WECP_WW1_E24hCW	Extractives	WW1 extract (24 h cold water)	–	–
WECP_WW2_24hCW		WW2 extract (24 h cold water)	–	–
WECP_WW3_E24hCW		WW3 extract (24 h cold water)	–	–
WCP_5%WW1*	Wood type	WW1	5	10
WCP_5%WW2		WW2	5	10
WCP_5%WW3		WW3	5	10
WCP_5%WW1*	Wood content	WW1	5	10
WCP_10%WW1			10	10
WCP_15%WW1			15	10
WCP_5%WW1*	Particle size	WW1	5	10
WCP_WW1_4mm			5	4

*Same sample

clarity's sake, wood-extractive cement paste (WECP) and wood cement paste (WCP) samples were identified as WECP/WCP_wt%WWi_hTT, where wt% is the relative amount of wood in weight, WWi is the wood type, h is the duration of pre-treatment in hours, and TT is the type of treatment.

Prismatic specimens of 160 mm × 40 mm × 40 mm with a water/cement ratio of 0.3 and cement pastes with a water/cement ratio of 0.5 were produced to perform the mechanical tests and hydration heat tests, respectively. The wood was previously ground, sieved and standardised by discarding particles that passed through the 1 mm sieve. The wood was then saturated for 24 h and the free surface water was discounted from the mix content. The water used for saturation was discarded, and tap water was used instead (except when studying the effect of wood extractives). The mix preparation and subsequent curing process followed the procedures of standard EN 1015-11:2019 for cement pastes.

Prior to the mechanical performance characterisation, the wood-cement pastes (WCPs) were evaluated for consistency in the fresh state through the flow table test as recommended in EN 1015–3:1999/A2:2006. Three parameters, namely, wood waste incorporation content, particle size gauge, and wood waste type, were chosen to assess their influence on the consistency of the WCP in the fresh state. Therefore, six WCPs, plus the reference, were selected to evaluate consistency.

A preliminary assessment of the possibility of developing a wood-cement concrete (WCC) was carried out next. Three concrete mixes were developed by partially replacing the mineral aggregates for the studied wood waste types. An ordinary Portland cement concrete composition was used as reference for comparison and aggregates replacement. To produce the reference concrete and the wood-concrete composites, three mineral aggregates were used: sand 0/4 (S0/4), gravel 0/5 (G0/5) and gravel 1 (G1). Portland cement CEM II/A-L 42.5 R, classified according to EN 197–1:2011 was used. The wood wastes were incorporated after 24 h submerged in water by replacing 25 v% the mineral aggregate (aggregate fractions were replaced based on the particle size distribution of each wood waste). The water absorbed by the wood was discounted from the contents. All wood-concrete composites also included a superplasticiser (Dynamon SP1, Mapei), used to improve the workability. The mix designs of the reference concrete and wood-concrete composites are detailed in Table 4.

2.2.1. Hydration heat evaluation

A hydration heat study was conducted using an isothermal calorimeter (I-Cal 4000 HPC, Calmetrix) to evaluate the influence of the wood on the heat of hydration of a cementitious paste at 23 °C. Performance calibration was conducted in accordance with the manufacturer's specifications.

The assessment was carried out by first evaluating the wood waste extractives' influence on the heat of hydration. This was done by producing cement pastes in which the water was replaced with wood waste extractives. The aqueous extractives were prepared with WW1, first soaking it in cold water (laboratory temperature), for 24 h and 24 h + 24 h, and then in hot water at 80 °C, for 2 h. Afterwards, WW1, WW2, and WW3 particles were incorporated in the cement pastes and the influence of the different wood waste types evaluated. Finally, the influence of the incorporation by weight (5%, 10% and 15%) for wood waste WW1 was also evaluated. Generally, the incorporation of wood fibres delays the cement setting time and decreases the hydration heat of cement, mainly because of the hydrolysis and solubilisation the wood

Table 4
Wood cement concrete (WCC) mixes (proportions in weight).

Mixture	Cement	Water	S0/4	G0/5	G1	Wood waste (dry)	Additive [1]
Reference	1.00	0.40	1.73	1.15	1.68	–	–
WCC_WW1	1.00	0.40	0.93	0.88	1.63	0.18 (WW1)	0.07
WCC_WW2	1.00	0.40	1.57	0.67	1.18	0.17 (WW2)	0.05
WCC_WW3	1.00	0.40	1.32	0.78	1.33	0.16 (WW3)	0.05

[1]Superplasticiser Dynamon SP1, Mapei

extractives [34]. In this context, wood waste WW1 was treated in cold water, hot water (80 °C) and a saturated solution of calcium hydroxide ($Ca(OH)_2$) prior to sample preparation, aiming to reduce wood extractives. The strategy of mineralising wood residues by treating the wood with sodium metasilicate (1 and 10%) was also exploited for WW1. Except for hot water treatment, in which the wood has been treated with hot water for two hours, wood waste was always extracted for 24 h with the different extractive solutions. The ratio of wood to the extractive solutions was 1:10 by volume. All wood waste incorporated in the cement pastes had prior cold-water extraction for 24 h, except for the pre-treatment analysis. A cement paste without wood or wood extractives was used as control.

To guarantee the cement content and a proper measurement temperature, the accurate amount of liquid was first measured and left for at least 2 h inside the calorimeter cell to stabilise at 23 °C. Meanwhile, cement was weighed, and the cement paste was prepared directly in the sample vial that was later placed inside the calorimeter cell. All samples had a fixed value of 40.00 g of cement and 20.00 g of water, under the range specified by the ASTM C1702-17; samples that had wood waste incorporated had the free surface water discounted from the added water. The samples were hand mixed for 30 s using a plastic spoon that was left inside the sample cup to prevent cement loss. The isothermal calorimeter measures the heat flow by means of a heat flow sensor, which is under the sample cup and in contact with a heat sink. To determine the heat of hydration of each sample, the measured thermal powers were integrated from the beginning to 48 h. Data were recorded per minute. The experimental design is detailed in Table 2.

2.2.2. Mechanical tests

To evaluate the mechanical properties of the wood-cement pastes (WCPs), flexural (3- point) and compressive strength tests were performed according to EN 1015-11:2019. An electromechanical universal testing machine, Instron model 59R5884 with a load cell of 10kN, was used in both tests. The load was applied without shock at a uniform rate of 10 N/s and 50 N/s, in the flexural and compressive strength tests, respectively. The compressive strength is determined from the two parts provided by the flexural strength test. For each mix, three prismatic specimens of 160 mm × 40 mm × 40 mm were produced to perform three flexural strength tests and six compressive strength tests.

Flexural strength, St_F, in N/mm^2, was determined according to equation $St_F = 1.5(F_F xl)/(bxd^2)$, where F_F is the maximal load, in N, l the span (distance between support rollers), 100 mm, and b and d correspond to the width and height of the specimen (both 40 mm).

Compressive strength, St_C in N/mm^2, was calculated by dividing the maximal load test, F_C in N, by the area of the cross section of the specimen, A (1600 mm^2).

To evaluate the mechanical properties of the wood-cement concrete (WCC) mixes, compressive strength tests were performed according to EN 12390–3:2019. The compressive strength σ, in N/mm^2, was assessed at 7 and 28 days for three cubic specimens from each composition at each age. A compression press (Controls, 50-C56V2) with a load cell of 3000 kN was used. The load was applied at a rate of 0.6 ± 0.2 MPa/s.

Tables 3 and 4 summarise the experimental design of WCP and the mix design of WCC, respectively.

3. Results and discussion

3.1. Chemical characterization (leaching tests)

Although there is no specific EU legislation regulating the use of end-of-life wood in construction products, it is common practice to screen for potential leachable substances that may represent a risk for the health and environment. As previously mentioned, taking into account the origin of the wood waste and the decade of production, it can be expected that the wood samples used in this work may contain creosote, CCA or ACQ preservatives.

Thus, to ascertain the nature of any preservatives and assess the amount of leaching substances, aqueous extracts of the different wood residues were produced according to EN 12457–2:2002. The aqueous extracts were then chemically analysed according to the procedures and criteria established for the acceptance of waste at landfills, originally established by the EU Council Decision 2003/33/EC [35] and later transposed into the law of the various Member States. Table 5 summarizes the results obtained, highlighting those exceeding the limits for the acceptance of waste in landfills for inert waste and non-hazardous waste.

Although most of the parameters surveyed did not have quantified, a few chemicals exhibited values above the limits of quantification (LOQ)

Table 5
Leaching results according to EN 12457–2:2002.

Parameter	LOQ	Wood sample			
		WW1	WW2	WW3	WW4
pH (25 °C)	—	4.6	7.1	5.1	5.3
Electric cond. 25 °C (mS/cm)	—	153	610	141	220
Chloride (mg/kg)	40	<LOQ	132	177	44
Fluoride (mg/kg)	4	<LOQ	<LOQ	<LOQ	<LOQ
Sulphate (mg/kg)	40	<LOQ	72	114	68
Phenol index (mg/kg)	0.50	2.2	3.8	1.2	827 *
As (mg/kg)	0.1	<LOQ	<LOQ	129 *	0.3
Ba (mg/kg)	2.0	<LOQ	<LOQ	<LOQ	<LOQ
Cd (mg/kg)	0.01	<LOQ	<LOQ	0.3	<LOQ
Cr (mg/kg)	0.2	<LOQ	<LOQ	21 **	<LOQ
Cu (mg/kg)	0.4	<LOQ	643 **	65 **	<LOQ
Hg (mg/kg)	0.01	<LOQ	<LOQ	<LOQ	<LOQ
Mo (mg/kg)	0.2	<LOQ	<LOQ	<LOQ	<LOQ
Ni (mg/kg)	0.1	<LOQ	<LOQ	<LOQ	<LOQ
Pb (mg/kg)	0.1	<LOQ	<LOQ	<LOQ	<LOQ
Sb (mg/kg)	0.02	<LOQ	<LOQ	0.1 *	<LOQ
Se (mg/kg)	0.02	<LOQ	<LOQ	<LOQ	<LOQ
Zn (mg/kg)	0.2	0.8	0.7	0.2	0.7
TOC (mg/kg)	250	502	1195	187	5814
Mineral Oil C10-C40 (mg/kg)	100	<LOQ	105	<LQ	1265 *
BTEX (mg/kg) [1]	0.15	<LOQ	<LOQ	<LOQ	<LOQ
PCB (mg/kg) [2]	0.35	<LOQ	<LOQ	<LOQ	<LOQ
PAH (mg/kg) [3]	16.0	<LOQ	<LOQ	<LOQ	43.1 *

[1] BTEX - Benzene, Toluene, Ethylbenzene, m-Xylene, p-Xylene, o-Xylene; [2] PCB - PCB 28, PCB 52, PCB 101, PCB 118, PCB 138, PCB 153, PCB 180; [3] PAH - Acenaphthene, Acenaphthylene, Anthracene, Benzo(a)anthracene, Benzo(a)pyrene, Benzo(b)fluoranthene, Benzo(g,h,i)perylene, Benzo(k)fluoranthene, Chrysene, Dibenzo(a,h)anthracene, Phenanthrene, Fluoranthene, Fluorene, Indene(1,2,3-cd) pyrene, Naphthalene, Pyrene; * Exceed the limits for the acceptance of waste in landfills for inert waste; ** Exceed the limits for the acceptance of waste in landfills for non-hazardous waste; LOQ - Limit of quantification, i.e. the smallest concentration of a substance that is possible to be determined by means of the respective analytical method.

of the methods (and, in some cases, above the acceptance limits of inert and non-dangerous landfill sites), confirming the presence of the chemical preservatives indicated in Table 1. As expected, no relevant heavy metals or organic compounds were detected in the eluate of the WW1 sample, assigned to untreated wood. The eluates of the WW2 and WW3 samples, respectively assigned to ACQ and CCA treated wood, revealed the presence of relevant heavy metals. In this case, only copper was detected in the eluate of the WW2 sample in a significant amount, while smaller amounts of several heavy metals (e.g. copper, arsenic and chromium) were detected in the eluate of the WW3 sample. As previously mentioned, the environmental risk of using wood particles containing heavy metals is low, since cement composites are found to fix such elements quite well [14]. Finally, relevant organic compounds given by the phenol index, mineral oil and PAH parameters were detected in the eluate of the WW4 sample, assigned to creosote treated wood.

Additional GC–MS studies were carried out to further investigate the composition of the eluates, concluding that a great variety of organic compounds were leached from WW4 sample, such as quinoline and its isomers, substituted quinolines, o-cresol and p-cresol, phenolic compounds and PAH (e.g. acenaphthylene, phenanthrene and naphthalene, etc.). The total ionic current (TIC) chromatogram for the eluate of the WW4 is presented in Fig. 2.

As expected, some organic compounds commonly found in virgin wood were also detected in the eluate of this sample (WW4), as well as in the eluates of the WW1 and WW2 samples. However, very low amounts of organic compounds were found in the eluate of the WW3 sample. A stacking representation of all chromatograms is shown in Fig. 3.

Given the nature and the amount of chemicals found in the eluate of the WW4 sample that represent a significant risk for the human health and environment, the authors decided not to proceed with the physical characterization of this wood waste and subsequent incorporation in the

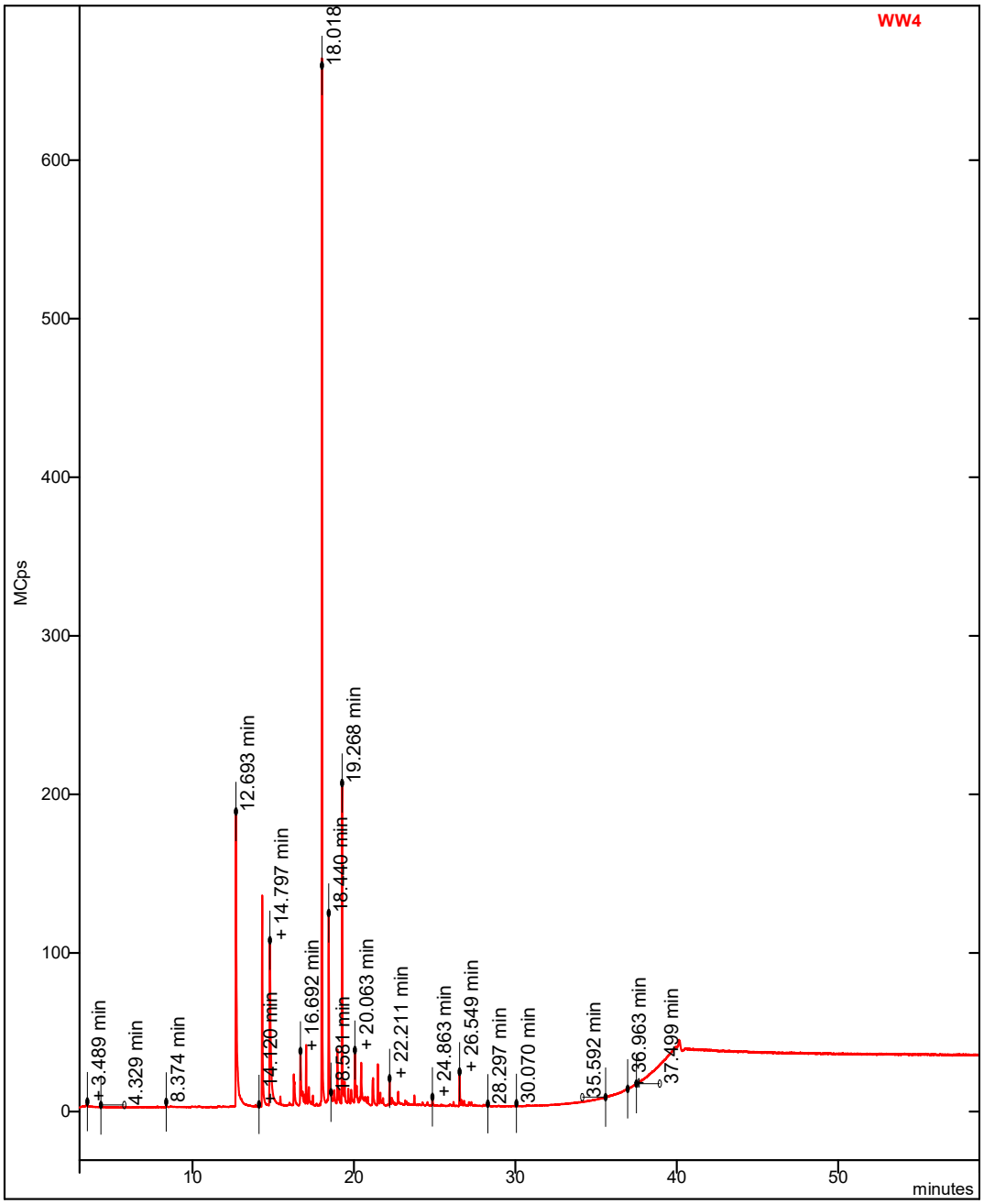

Fig. 2. Total ionic current (TIC) chromatogram for the eluates of the WW4 sample.

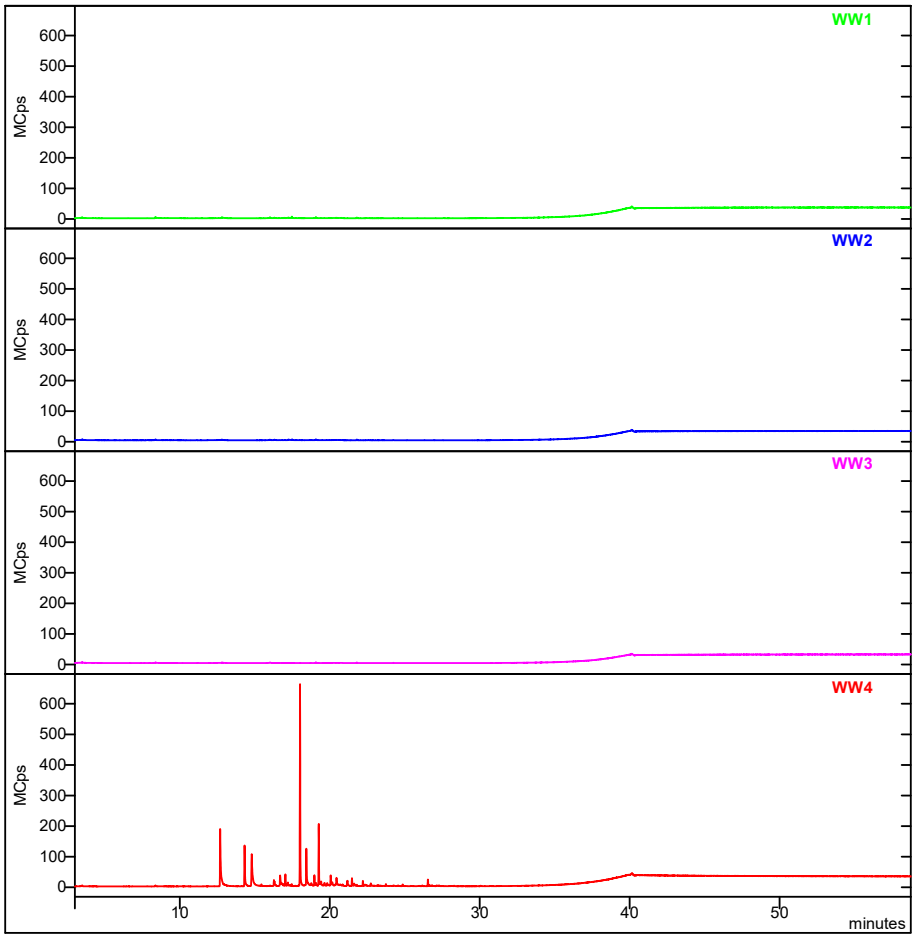

Fig. 3. Stacking representation of the total ionic current (TIC) chromatograms for the eluates of the WW1, WW2, WW3 and WW4 samples.

cement composites. As noted earlier, some of the compounds found in creosote-preserved wood have been linked with harmful effects to human health.

3.2. Physical characterisation

Besides the chemical aspects, the design of innovative composites also requires a complete physical characterisation of the wood waste. This should cover, for example, water absorption capacity and density, and it is stressed that particle size can have a significant influence on these properties.

Thus, each wood waste type was physically characterised in terms of density, water absorption and free surface water by adapting the procedures of the European Standard EN 1097-6:2013.

The three as-received wood waste types were placed in a drying oven at 105 °C for 24 h to dry, after which they were grinded into small particle size using a powder grinder and a screen. The influence of the particle size gauge was studied for the virgin wood (WW1) ground with a 4 mm and 10 mm screen. The other two wood wastes were studied for only one particle size (10 mm screen).

The wood waste physical characterisation results are given in Table 6 and the water absorption is shown in Fig. 4.

The results show that a substantial amount of water is absorbed by the wood fibres or dragged in the surface after immersion (free surface water). It also shows that the total water content vary significantly between the untreated wood sample (240%) and treated wood samples (214 % and 215 %) for the same particle size (10 mm), which could be related to the micropore structure of the different wood waste samples [36]. Additionally, as expected, an even greater effect is observed when particle size changes from 10 mm (240%) to 4 mm (290%), which is likely to occur due to the easier water intake considering the larger surface area of the wood particles.

The results of water absorption over time further shows that although most water is absorbed after 2 h (greater than 90%), more time may be needed to achieve complete hydration of the wood fibre. Note that during the production of cement composites, incomplete hydration of the fibre may result in competition for water.

Table 6
Wood waste physical characterisation.

Wood waste type	Maximum size (mm)	Density (kg/m^3)		Water content (%)		
		Oven dried	Saturated with dry surface	Water absorption	Free surface water	Total
WW1	4	280	1070	239	54	293
	10	380	1090	193	47	240
WW2	10	390	1060	154	61	215
WW3	10	390	1070	147	67	214

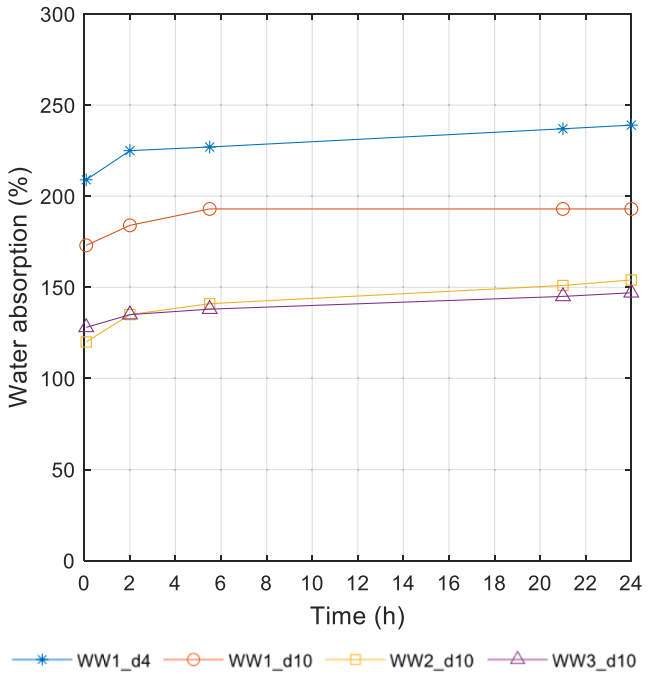

Fig. 4. Water absorption by different wood waste type (WW1, WW2 and WW3) and wood particle size (4 mm, 10 mm) over time.

3.3. Hydration heat evaluation of WCP

Since monitoring the hydration heat of cement is one of the best ways to check its chemical compatibility with different additions, 15 different cement pastes containing wood extractives and wood particles were produced as previously described and the hydration heat over 48 h was evaluated against a control mix. For the purposes of the study, variations were introduced in terms of the type of wood (WW1, WW2 and WW3), the wood content (5 w%, 10 w% and 15 w%) and the pre-treatment of wood (cold water extraction, hot water extraction, calcium hydroxide extraction and sodium silicate mineralization). The results are summarised in Table 7.

As mentioned above, a control mix was analysed resulting in a 48 h HoH of 243.9 J/g of cement. Regarding the extractive influence, the pastes does not show a significant 48 h HoH reduction in comparison with the control mix, with the WECP_WW1_E24hCW, WECP_WW1_E48hCW and WECP_WW1_E2hHW pastes showing respectively 3%, 2% and 1% of 48 h HoH reduction. Note that a study carried out with the control sample for 6 replicas recorded a standard deviation of 1.3 J/g with a variation coefficient of 0.6%. However, according to EN 196–11:2018, two results for the same sample should not differ by more than 10 J/g, a value only slightly exceeded for 10% and 15% incorporation of wood particles. A setting time delay was also observed for all the cement pastes, with WECP_WW1_E2hHW presenting the highest impact (≈ 2 h). These results can be explained since some of the wood extractives are expected to generate a delay in the setting time without significantly affecting the extent of HoH. Fig. 5 shows the HoH (left) and the thermal power (right) plots for pastes containing different wood extracts from that in the control.

Regarding the waste type, WCP_5%WW1 had the worst performance by reducing the 48 h HoH by 4% relative to the control mix, while WCP_5%WW2 and WCP_5%WW3 reduced it by only 3%. WCP_5%WW1 also recorded the longest setting time, nearly 1 h. Both results were expected because WW2 and WW3 had been leached during their service life, as opposed to WW1. Furthermore, the preservative treatments could be responsible for reducing the release of undesired wood extractives. Fig. 6 shows the HoH development (left) and the thermal power progress (right) for pastes containing different waste type in comparison with the control.

Table 7
Heat of Hydration (HoH) after 48 h for the different wood-cement pastes (WCPs).

Sample reference	Parameter	48 h HoH		Setting time (h)
		HoH (J/g)	Reduction (%) relative to the control sample	
Control	–	243.9	–	6.67
WECP_WW1_E24hCW	Extractives	235.8	3	7.38
WECP_WW1_E48hCW		240.1	2	6.97
WECP_WW1_E2hHW		240.9	1	8.28
WCP_5%WW1*	Wood type	233.3	4	7.68
WCP_5%WW2		236.0	3	7.50
WCP_5%WW3		236.3	3	7.43
WCP_5%WW1*	Wood content	233.3	4	7.68
WCP_10%WW1		216.6	11	9.07
WCP_15%WW1		209.6	14	9.63
WCP_5%WW1_4hCW	Wood pre-treatment	230.1	6	7.83
WCP_5%WW1_6hCW		231.4	5	7.90
WCP_5%WW1_8hCW		232.1	5	7.94
WCP_5%WW1*		233.3	4	7.68
WCP_5%WW1_2hHW		235.2	4	7.97
WCP_5%WW1_24hCH		231.3	5	7.37
WCP_5% WW1_24hSS1%		234.1	4	7.68
WCP_5% WW1_24hSS10%		237.6	3	9.17

*Same sample

The HoH results from the wood incorporation by weight decreased significantly, based on the incorporation increase where WCP_5%WW1, WCP_10%WW1 and WCP_15%WW1 respectively saw 4, 11, and 14 % reductions in HoH. Note that a greater effect of the wood particles on the cement paste was expected, compared with the wood extractives, considering the liquid/solid ratio used to produce the eluates. In terms of setting time, a significant time increase was also noted where WCP_15%WW1 took almost 3 h longer than the control and almost 2 h more than WCP_5%WW1. The increase of wood content also increases the disturbance and the extractive content in the cementitious matrix, thereby justifying the results. Fig. 7 shows the HoH (left) and the thermal power plots (right) for pastes with different wood contents from the control.

Although the wood pre-treatments did not lead in general to any significant improvement in the HoH performance of the cement pastes, a slight improvement was exhibited by WCP_5%WW1_24hSS1%, WCP_5%WW1_24hSS10% and WCP_5%WW1_2hHW compared with WCP_5%WW1. Comparing now WCP_5%WW1_4hCW, WCP_5%WW1_6hCW and WCP_5%WW1_8hCW, it was found that the cold-water extraction time has little influence on the results of the HoH. Apart from the impact on the HoH, WCP_5%WW1_2hHW, WCP_5%WW1_4hCW, WCP_5%WW1_6hCW and WCP_5%WW1_8hCW had a setting time similar to that identified in WCP_5%WW1, which emphasises that the impact of the presence of particles in disturbing the cementitious matrix is greater than that of the wood extractives. Besides having improved HoH performance, WCP_5%WW1_24hSS10% recorded an almost 2 h longer setting time than the other mixes. Fig. 8 shows the HoH (left) and the thermal power (right) evolution for pastes containing wood with different pre-treatments, compared with the control.

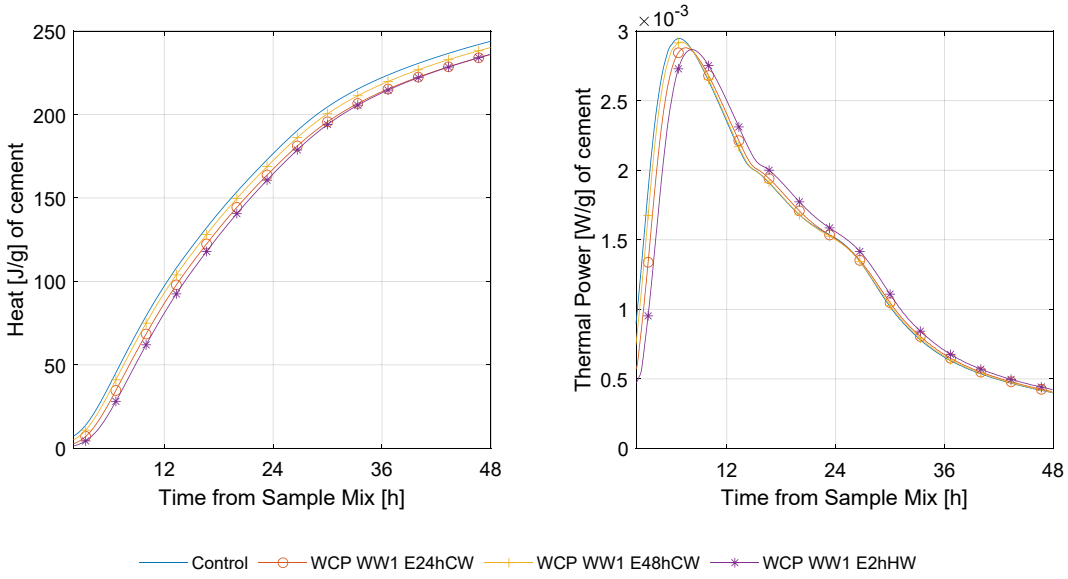

Fig. 5. Evolution of HoH (left) and thermal power (right) for different wood-cement pastes (extractive influence).

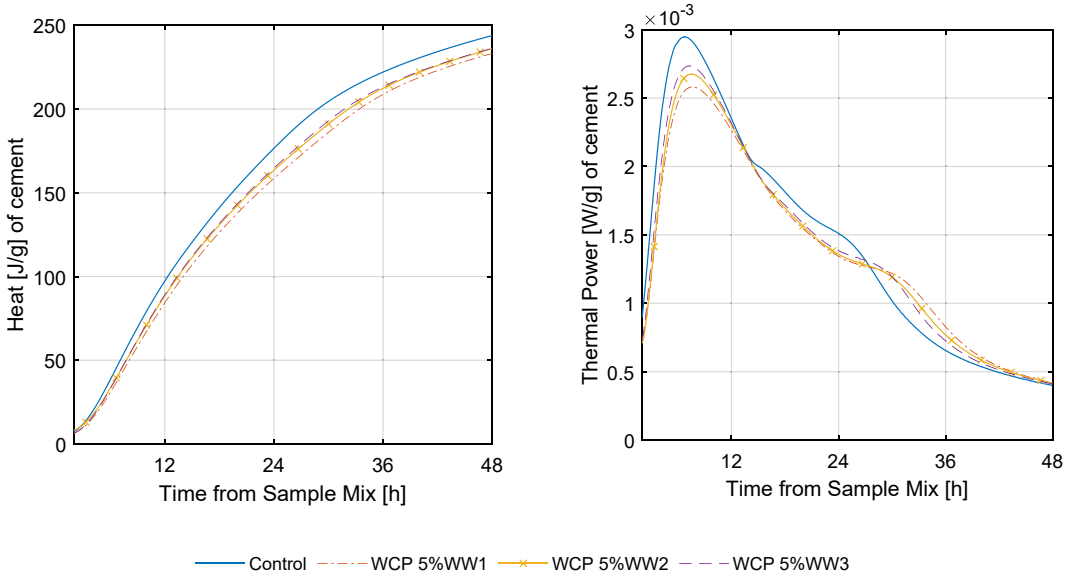

Fig. 6. Evolution of HoH (left) and thermal power (right) for different wood-cement pastes (wood type influence).

3.4. Mechanical properties of WCP

To further study the effect of wood waste incorporation on the mechanical properties of cement composites, nine cement pastes containing both wood extractives and wood particles were produced as previously described. For the purposes of the study, variations were introduced in terms of the type of wood (WW1, WW2 and WW3), the wood content (5 w%, 10 w% and 15 w%) and wood particle size (4 mm and 10 mm).

The consistency of six WCPs was determined by the flow table test in the fresh state during the prismatic specimen preparation (water/cement ratio of 0.3). The consistency results from the wood waste incorporation content showed a decrease in the consistency with increased incorporation of wood in the mix, which can be related to the organisation of the fibres into the cementitious matrix. This behaviour is similar to a consistency assessment made by researchers who replaced cement with sawdust in up to 5 w% [37]. However, compared with the reference paste, WCP_5%WW1 showed a small increase in consistency. When evaluating the wood particle size, WCP_5%WW1_4MM showed a significant increase in flowability compared with WCP_5%WW1 (10 mm) and with the reference. The water absorption behaviour of the wood fibres could explain this characteristic and the lower consistency recorded for WCP_5%WW2 and WCP_5%WW3. On the one hand, the higher water absorption of the 4 mm gauge WW1 can lead to water being released into the mix because of the heat generated in the initial hydration of the cement. On the other hand, the low water absorption of WW2 and WW3 can justify the differences found in the results when different wood waste types were incorporated (WCP_5%WW1, WCP_5%WW2 and WCP_5%WW3). The results of the consistency test are presented in Table 8.

The results of the mechanical tests are presented in Figs. 9 and 10. The compressive strength results show that the incorporation of wood fibres leads to a considerable reduction in compressive strength relative to the reference. It seems, however, that the main impact is related to the wood incorporation content, while only small variations were found when changing the wood waste type and particle size. Reductions of

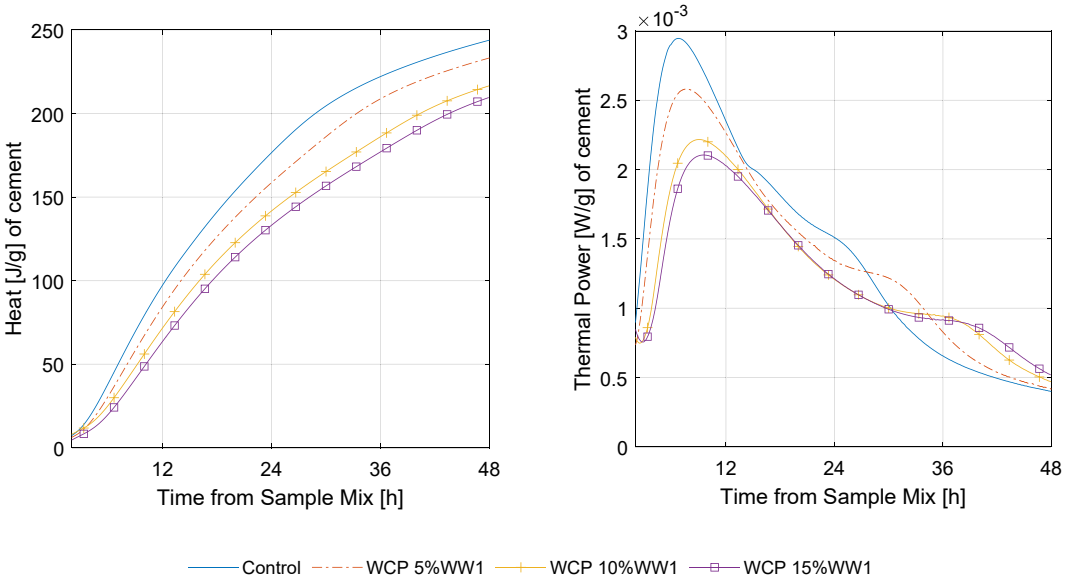

Fig. 7. Evolution of HoH (left) and thermal power (right) for different wood-cement pastes (wood content influence).

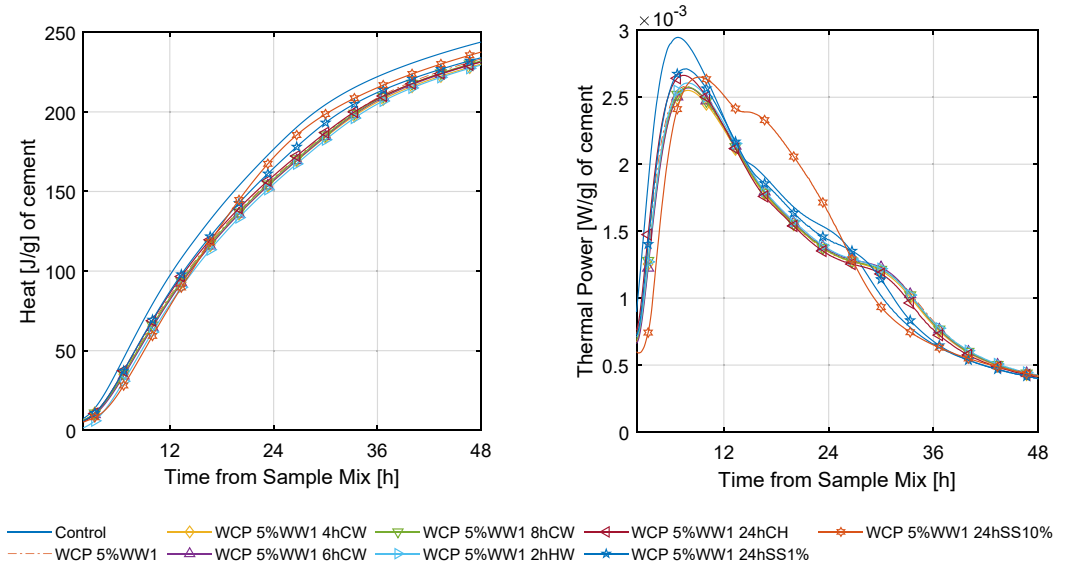

Fig. 8. Evolution of HoH (left) and thermal power (right) for different wood-cement pastes (wood pre-treatments influence).

Table 8
Consistency in fresh state (flow table test).

Paste	Consistency (mm)	Variation (%) relative to the reference sample
Reference	193 ± 2	–
WCP_5%WW1	203 ± 3	5
WCP_10%WW1	193 ± 0	0
WCP_15%WW1	165 ± 2	15
WCP_5%WW1_4MM	218 ± 2	13
WCP_5%WW2	189 ± 1	2
WCP_5%WW3	187 ± 5	3

43%, 64% and 80% of compressive strength at 28 days were found for WCP_5%WW1, WCP_10%WW1 and WCP_15%WW1, respectively. A similar work that studied mortars containing not only cement but also fine aggregates, reports that adding sawdust to replace 5 w% of cement, which is nearly 2 w% of the sample total mass, led to a reduction of 28% in compressive strength [37]. Other researchers assessed wood-cement pastes in which cement was replaced with sawdust in amounts of around 9%, 17% and 23 w% found that compressive strength fell by 49%, 57% and 76 % at 28 days, respectively [38]. However, smaller effects were reported on the mechanical properties of wood-cement boards when using wood ashes as a partial replacement for cement up to 30% [32].

Although small variations can be seen between the different samples, the high variability of the flexural strength results means that conclusions cannot be drawn on the actual effect of wood incorporation. This could be caused by the heterogeneous dispersion of wood particles measuring up to 10 mm, when test specimens of 160 mm × 40 mm × 40 mm are used. Additionally, the percentage of volume occupied by wood particles is significant, even with low incorporation of wood (Fig. 11). Researchers who carried out a similar study with a mortar containing nearly 2 w% of sawdust in the sample total mass, recorded a reduction of

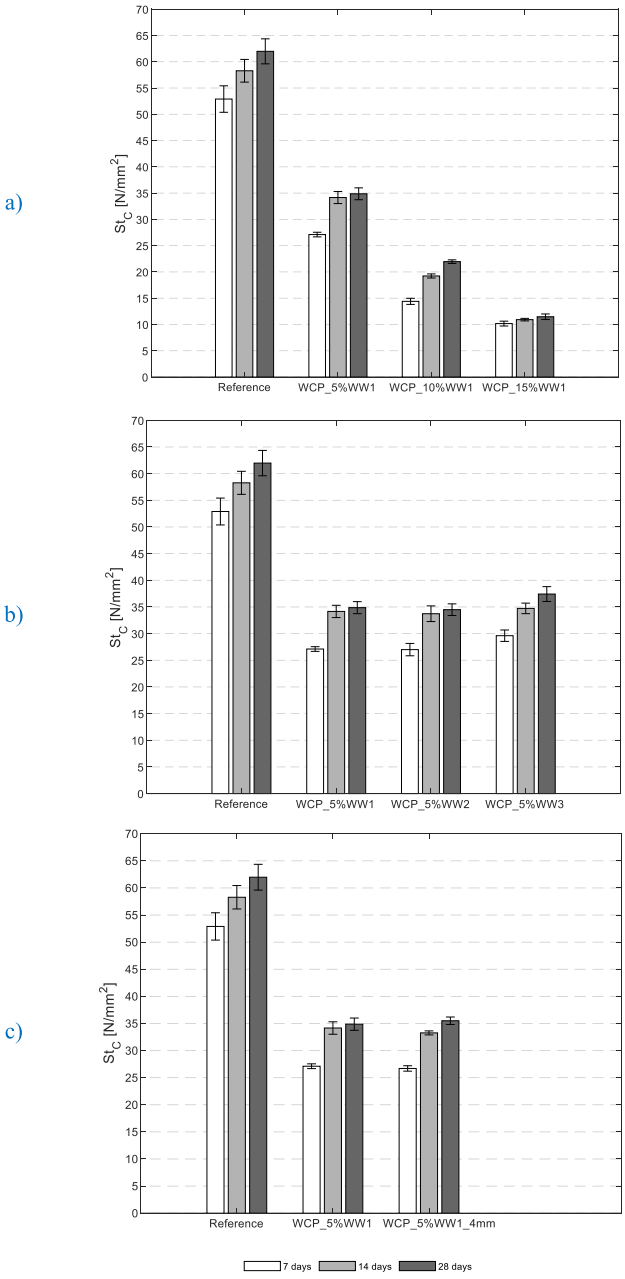

Fig. 9. Compressive strength, St_C, mean results (± standard deviation): a) Wood waste content; b) Type of wood waste; c) Maximum size.

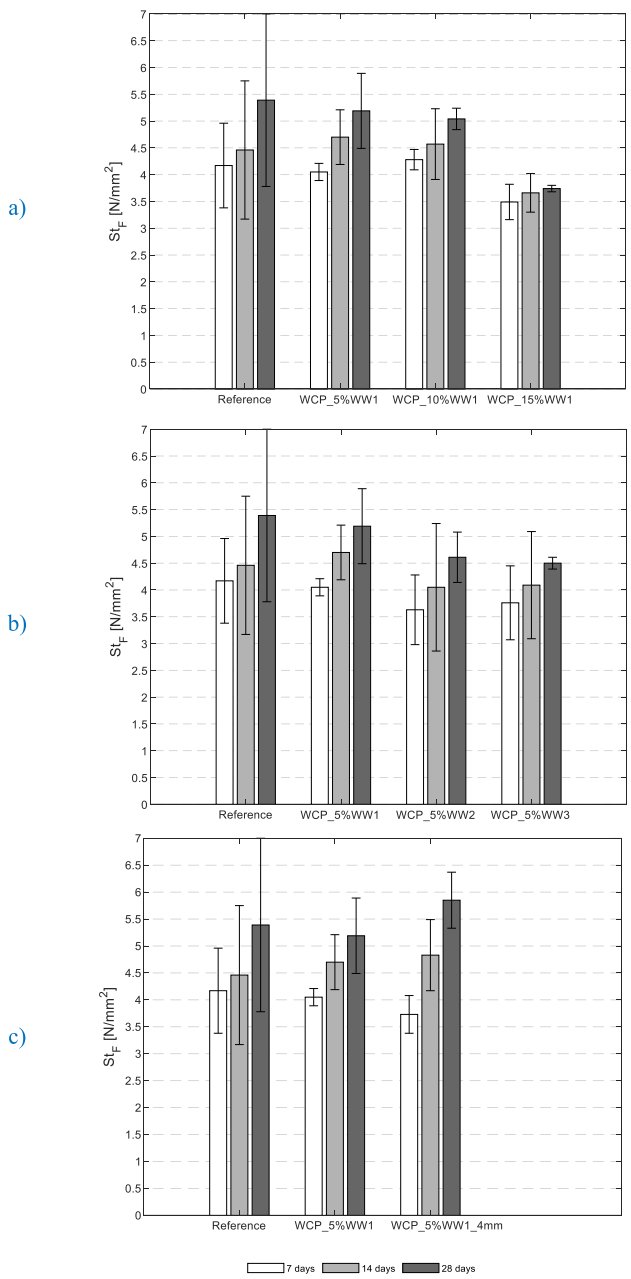

Fig. 10. Flexural strength, St_F, mean results (± standard deviation): a) Wood waste content; b) Type of wood waste; c) Maximum size.

20% in flexural strength [37]. This decrease could be related directly to the cement reduction differing from the present work, which had no inert besides the wood waste.

3.5. Mechanical properties of WCC

The compressive strength of the wood-cement concrete has been assessed. This highlighted that, like WCP, the incorporation of wood in the concrete significantly reduced the mechanical performance of the composites. When the composites were being prepared, it was clear that the workability of the mixes was poor and compromised the composite production and strength development. This can be seen from the voids distributed in the cubic specimen and the mass of the samples. Therefore, it is important to analyse the compressive strength results not only in terms of mean value, but also related to their respective density. Mean values of density and compressive strength for the reference mix and wood-concrete composites are presented in Table 9.

The WCC, where the mineral aggregates were partially replaced with wood waste in 25 v%, had a compressive strength reduction in the range of 38–50% and 34–48% at 7 and 28 days, respectively. Although the strength development could be partially influenced by the superplasticizer content, the compressive strength reduction seems to be more related to WCC density than to the waste type. As Fig. 12 shows, an almost linear relationship between the composites' density and compressive strength is obtained, with coefficient of determination (R^2) of 0.982 and 0.998 at 7 days and 28 days, respectively.

Similar results have been reported in other studies. A wood-cement concrete with 15 v% of fine aggregates being replaced with sawdust led to a compressive strength reduction of 42% and 40% at 7 and 28 days, respectively [39]. A compressive strength reduction of 63% and 64% at 7 and 28 days, respectively, have been observed for a WCC where fine aggregates were replaced with sawdust in 20 w% [40].

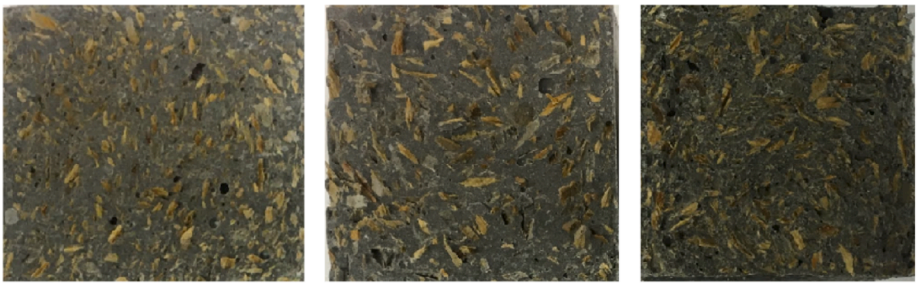

Fig. 11. WCP test specimens' cross-section images and percentage of area occupied by the wood fibres calculated with Image J software (version 1.52a).

Table 9
Wood-cement concrete (WCC) mixes: density and compressive strength.

Mix	Harden density water-saturated (kg/m^3)		Compressive strength (MPa)	
	7 days	28 days	7 days	28 days
Reference	2403 ± 11	2426 ± 18	58.0 ± 0.4	64.3 ± 4.0
WCC_WW1	2202 ± 29	2230 ± 15	34.8 ± 3.1	42.3 ± 1.2
WCC_WW2	2104 ± 8	2121 ± 81	28.9 ± 1.5	33.0 ± 8.6
WCC_WW3	2184 ± 24	2157 ± 30	35.7 ± 1.9	36.6 ± 4.2

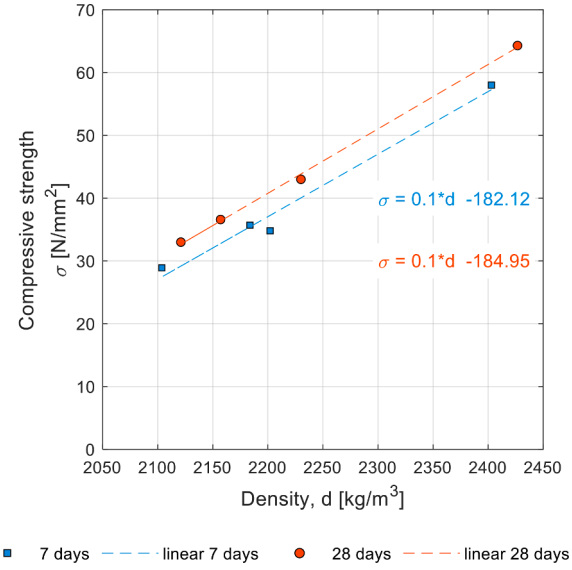

Fig. 12. Compressive strength of wood-cement concrete (WCC) mixes as a function of density.

The incorporation of wood waste does not limit the application of these composites to non-structural applications, since the compressive strength is above 25 MPa. Although there are promising aspects in this domain, further studies are needed to explore the advantages of these composites, especially in terms of physical performance (hygrothermal and acoustic), weight reduction, durability, and sustainability.

4. Conclusions

The practical feasibility of using end-of-life treated wood for producing innovative lightweight cement composites was investigated. This included a characterisation of wood residues from different sources to assess the presence of leaching substances and determine the physical properties relevant to the production of cement composites. It also included the production of cement pastes containing different wood waste particles for determining the hydration heat and mechanical properties as a function of wood waste origin, wood content, wood particle size and wood pre-treatment. Concrete mixes containing selected wood particles were also produced for a preliminary examination of the effect of wood waste on the mechanical properties of a reference concrete.

The results show that the concentration of leaching substances can vary significantly depending on the source of wood waste, exceeding in some cases the acceptance limits for inert or non-hazardous waste landfills. As expected, no relevant chemicals were detected in the eluate of the WW1 sample, assigned to untreated wood. For their part, the eluates of the WW2 and WW3 samples, respectively assigned to ACQ and CCA treatments, revealed the presence of significant amounts of some heavy metals (e.g. copper, arsenic and chromium). Finally, relevant organic compounds were detected in the eluate of the WW4 sample, assigned to creosote treated wood. However, while the environmental risk of using wood particles from WW2 and WW3 sources is low, since cement composites are likely to fix such heavy metals quite well, wood particles from the WW4 source poses a higher risk because such organic compounds are not expected to bind cement matrixes in an efficient manner.

Although small variations were found in the hydration heat profile of different cement composites containing up to 5 %(w/w), the incorporation of increasing quantities of wood particles in cement pastes leads to a more significant reduction in the HoH released. Small variations were also found when changing the types of wood waste and pre-treatments. As expected, untreated wood from sawmills (WW1) affected the hydration heat profiles slightly more than both the treated woods containing preservatives from agriculture posts and fences (WW2, WW3), which is plausible taking into account the leaching process of the latter over the service life.

The results also show that a significant reduction of compressive strength was observed with increasing percentages of wood particles, which can be attributed to the greater impact of the wood particles in disturbing the cementitious matrix in comparison to that of the wood extractives. The incorporation of wood particles also resulted in a significant reduction of the mechanical performance of the hardened material, though the use of such composites in applications with low-structural requirements has not been limited.

CRediT authorship contribution statement

Sara Dias: Investigation, Writing – original draft. **João Almeida:** Conceptualization, Methodology, Validation, Writing – original draft. **Bruna Santos:** Investigation, Writing – original draft. **Pedro Humbert:** Investigation, Methodology, Writing – original draft. **António Tadeu:** Conceptualization, Methodology, Validation, Writing – review & editing, Funding acquisition. **Julieta António:** Conceptualization, Methodology, Validation, Writing – review & editing. **Jorge de Brito:**

Validation, Writing – review & editing. **Pedro Pinhão:** Investigation.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Acknowledgements

This study was supported by the Portuguese Foundation for Science and Technology (FCT), under PhD grant PD/BD/150575/2020, as part of the EcoCoRe Programme. It was also supported by the Project Prock (CENTRO-01-0247-FEDER-039790) funded by Portugal 2020 through the Operational Programme for Competitiveness Factors (COMPETE2020). The authors are also grateful to Sabril - Sociedade de Areias e Britas, Lda, Secil Group, and Mapei Portugal which kindly supplied the materials used in this study: wood waste, mineral aggregates and cement, and adjuvant, respectively.

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