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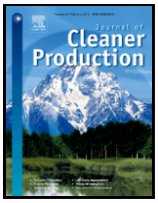

Mine waste as a sustainable resource for facing bricks

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ABSTRACT

Circularity of raw materials starts with a zero-waste approach to any (potential) resource. The increasing pressure on the primary raw materials sector and external market dependence can ultimately lead to scarcity of natural resources. To minimise this pressure, alternative materials need to be mapped, characterised, and valorised. Construction and demolition waste and mining waste are currently the biggest waste streams in the EU27 and can lead to environmental, health, and social hazards. The purpose of this study is to evaluate the sustainable use of (clean) sulphidic mining waste materials in facing bricks.

After materials' characterisation, a company-specific blend for facing bricks has been modified on a lab scale, by partly replacing primary raw materials with mining waste materials (Plombières tailings and Neves Corvo waste rocks). The production processes, product quality, and environmental performance of waste-derived bricks were assessed and compared to standards and regulatory limits. A cradle-to-gate life cycle assessment (LCA) was performed to evaluate the environmental profiles of the best performing waste-derived bricks compared with standard bricks.

Results: show the suitability of using the untreated Plombières tailings material in facing brick blends, giving satisfactory technical and aesthetical properties, and complying with environmental regulations for service, 2nd life, and end-of-life stages. According to the LCA, facing bricks made with 40 wt% Plombières tailings demonstrated better environmental performances than standard bricks. The pre-treated Neves Corvo waste rock materials were unsuitable for facing bricks due to the high sulphur and metal(loid)s content that caused aesthetical and chemical-environmental problems.

1. Introduction

The global use of materials, especially metals and non-metallic minerals, is expected to more than double by 2060, with one-third of this rise coming from the building and construction sector demand (OECD, 2019). This usage will continue to increase due to the continuous technological development of thriving societies and the urban development aligned with the global population growth and green energy transition (Buchholz and Brandenburg, 2018). The natural resources needed for building and construction purposes, especially non-metallic minerals such as sand and gravel, can be in global scarcity by 2050 (Sverdrup et al., 2017). In 2017, non-metallic mineral deposits accounted for almost half (48%) of the global natural resource extraction (IRP, 2019). Moreover, natural resource exploitation, such as sand mining from river beds, is leaving a negative footprint on the ecological, health, and social aspects of nearby populations (Best, 2019; Bendixen et al., 2019).

Using existing waste streams (e.g., mining waste or construction and demolition waste) can help reducing the pressure on natural resources exploration and accelerate the shift towards a more circular economy. In Europe, construction and demolition waste (CDW) represents the biggest waste stream in volume (36%), followed by mining and quarrying waste (27%), together accounting for more than half of the total waste output in the EU27 (Eurostat, 2018). Setting up a (waste) materials database including all of the intrinsic properties and potential applications for waste streams should be a priority to mitigate value loss and, most importantly, mitigate environmental, health, and social hazards associated with waste disposal. Sulphidic tailings and waste rocks are considered as challenging side streams due to the potential in generat-

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ing acid mine drainage (AMD) when in contact with oxygen, water, and micro-organisms (Rodríguez-Galán et al., 2019). Hazardous metal (loid)s and sulphates are leached at low pH and can end up in soils and aquatic environments, causing a severe ecological impact (Kefeni et al., 2017). Finding a suitable solution for the metal (loid)s and sulphur in these sulphidic mining waste materials and the (bulk) mineral residual fraction, generally rich in quartz and clay minerals, is key for a more sustainable approach within the raw materials sector.

The use of mining and quarrying waste in ceramics, especially ceramic bricks, has been broadly studied (Murmu and Patel, 2018) not only due to stricter European environmental and land-use regulations but also due to the need for a more resource efficient economy. Nevertheless, the use of sulphidic mining waste streams in traditional building ceramics, such as fired bricks, can cause technical, aesthetical and, especially, environmental problems because of their significant sulphur and hazardous metal(loid)s content. Many studies only focus on technical and/or aesthetical properties without considering environmental compliance, which is fundamental to place a waste-derived ceramic product in the market. Roy et al. (2007) assured the required technical criteria for compressive strength, linear shrinkage and water absorption of brick made with 45 wt% of gold tailings and 55 wt% of red soil. Moreover, the cost of these soil-tailings bricks would be 85% of the cost of conventional bricks. Yonggang et al. (2011) showed that bricks containing 75 wt% of fine gold tailings and 25 wt% of clay mixtures achieved a compressive strength of MU10, with no lime blowing or efflorescence. Chen et al. (2011) obtained bricks with satisfying technical properties in compliance with Chinese standards for bricks containing a 84:10:6 ratio of hematite tailings, clay, and fly ash, respectively. Salinas-Rodríguez et al. (2017) showed that 55–65 wt% of Au–Ag tailings can be used as feldspar substitutes in ceramic brick mixtures giving similar or better technical properties than those of conventional bricks. Suvorova et al. (2017) investigated the production of bricks containing Cu–Ni tailings (40 wt%), apatite-nepheline tailings (40 wt%) and ferriferous quartzite tailings (20 wt%). Results showed satisfactory technical performance (frost resistance) for samples fired at 900 ˚C and 950 ˚C, making them a potential good fit as high-quality full and hollow bricks. The production of iron ore tailings bricks with a hydrous foam-gel casting method, resulted in a high strength and low thermal conductivity, making the porous bricks suitable for building thermal insulation (Li et al., 2019). Mendes et al. (2019) incorporated iron ore tailings in clay-based bricks that after firing at 850 ˚C for 3 h. The bricks showed satisfactory mechanical properties, decreased water absorption, apparent porosity, and firing shrinkage, but lower strength due to lack of clay minerals. Wei et al. (2021) used gold tailings and clay as an additive at 35%, with moisture content of 25% and firing temperature of 1030 ˚C for 105 min, which gave optimal process parameters, such as compressive strength.

In none of these studies, the environmental performance of the bricks was addressed. On the one hand, using mine waste as an alternative raw material allows to save primary resources. On the other hand, dealing with mining waste materials with high sulphur concentrations will lead to efflorescence and the formation of a black core in the ceramic bodies, as well as high SO_x emissions (Veiga Simão et al., 2021a) and poor environmental performance of the fired bodies due to excessive leaching of hazardous metal (loid)s (Taha et al., 2017; Belmonte et al., 2018). Pre-treatment possibilities for these alternative materials (e.g., physical, thermal, and/or (bio)chemical treatment) should also be envisaged, if necessary, before valorisation routes can be proposed. In a recent study the feasibility of incorporating untreated sulphidic tailings in ceramics was demonstrated. This study showed that low sulphidic Pb–Zn tailings could, without any pre-treatment, be directly used in blends for roof tiles (5 wt%), blocks (10 wt%), and pavers (10 and 20 wt%) giving satisfactory technical, aesthetical, and chemical properties of the fired bodies, as well as compliance with environmental regulations for 2nd life usage, where demolished tailings-containing ceramics can be recycled as aggregates (Veiga Simão et al., 2021b). Another recent study showed that low sulphidic Pb tailings could be used in ceramic bricks (up to 90 wt%) with satisfactory physical and mechanical properties, as well as a compliant active soluble salt content (Suárez-Macías et al., 2020).

Life cycle assessment (LCA) is a frequently used method to assess the environmental performance of products, processes, or services. For products, LCAs evaluate the overall environmental performance from the extraction of raw materials until the product leaves the factory (gate) or until the product demolition and end-of-life (grave). All relevant processes, resource extractions, and environmental emissions are accounted for and assessed in the life cycle impact assessment stage to quantify, e.g., impacts on climate change or toxicity of the product. Among life cycle impact assessment methods for characterising different impacts, LCAs can follow particular guidelines, such as applying LCA for the Environmental Footprint Initiative of the European Commission (Manfredi et al., 2012). Examples of LCA studies dealing with waste-containing ceramic bricks can be found in a recent review article (Huarachi et al., 2020), where 28 studies were analysed, suggesting that environmental benefits can be achieved by reusing waste streams from various industries, resulting in a set of alternative bricks made from organic or inorganic additives (Bories et al., 2016; Huang et al., 2017; Lozano-Miralles et al., 2018; Muñoz et al., 2018).

The current study presents a holistic approach for assessing the potential valorisation of sulphidic mining waste materials, replacing up to 40 wt% of virgin raw materials, in facing bricks not only considering production processes (shaping, drying and firing), and product quality (technical, aesthetical, and chemical properties), but also environmental performance (leaching of metal (loid)s) during service life, second-life, and end-of-life stages). Moreover, a cradle-to-gate life cycle assessment (LCA) was performed to assess environmental benefits when comparing waste-derived bricks with standard bricks.

2. Materials and methods

2.1. Materials

2.1.1. Mining waste

The former Plombières Pb–Zn mining site (Eastern Belgium), inactive since 1922 (Dejonghe et al., 1993), contains 11.4 Mt of mining waste that is stored in tailings ponds covered with soils and metallurgical waste (Helser and Cappuyns, 2021). In a tailings pond, a sample of around 25 kg (PL_62_I) was taken at 1.10 m of depth from a hand-excavated pit hole and stored in sealed containers. The sample comes from a Pb–Zn poor yellow tailings layer, representing 73 wt% of the total quantity of mine tailings and metallurgical waste from that tailings pond (Bevandić et al., 2021).

The active Neves Corvo Cu–Zn mine (Southern Portugal) has been operating since 1988, continuously producing copper, tin (exhausted), zinc, and lead concentrates from five massive sulphide orebodies (Escobar et al., 2021). In the last decade (2010–2019), 7.3 Mt of fresh waste rock material (sample NC_01) was generated at Neves Corvo Cu–Zn mine and co-deposited with thickened tailings in a tailings management facility. Additionally, 3.1 Mt of old and oxidised waste rock material was stored in open-air temporary stockpiles (sample NC_03) (Escobar et al., 2021). For this study, both Neves Corvo waste rock materials were cleaned by froth flotation in order to remove their high sulphidic and meta (loid)s, which led to technical and aesthetical problems for ceramics (Veiga Simão et al., 2021a). About 20 kg of cleaned Neves Corvo fresh (NC_01_CL_FLOT) and stored (NC_03_CL_FLOT) waste rock materials were shipped to Belgium in sealed containers.

2.1.2. COMPANY-SPECIFIC raw materials, ceramic blends and lab test specimens

The primary raw materials used to produce one company-specific facing brick blend (LM) included local loam, imported clay A, imported clay B, and imported filler. Around 50 kg of each primary raw material was sampled at the production plant in Belgium and stored in sealed containers.

The standard and six modified facing brick blends were produced on a lab scale using a blending machine and 15 lab test specimens per blend were produced using a hand-mold press. The production processes are described in section 2.2.2.

2.2. Methods

2.2.1. Sample pre-treatment

The Plombières yellow tailings material (PL_62_I) is a fine grained material (Appendix A) with low content of sulphur and metal (loid)s (Table 1A), which makes it ready-to-use in the proposed facing brick blends. Therefore, Plombières tailings material was only dried overnight in a ventilated stove (Heraeus UT 6060) at 105 °C. The original Neves Corvo fresh (NC_01) and stored (NC_03) waste rock materials needed pre-treatment before incorporation in ceramic blends (Veiga Simão et al., 2021a). Froth flotation was chosen to clean both waste rock materials from sulphur and hazardous metal (loid)s. The grain size of the waste rock material was reduced by a custom made comminution system, which included dry horizontal sieving (9 and 2.5 mm), double roll crushing with sieving (2.5 mm), drying (45 °C for 4h) and grinding in a ball mill (20–30 min, 55 rpm), wet horizontal sieving (87 μm), settling and dewatering, wet grinding in a ball mill (30 min, 55 rpm), and lastly settling and de-watering. After reaching a fraction of <65 μm, both waste rock materials were cleaned following flotation parameters normally used for sulphidic ores, including potassium amyl xanthate (KAX) collector at 50 g/t, methyl isobutyl carbinol (MIBC) frother at 100 g/t, pH 9, and solid/liquid ratio of 300 g/l. After flotation, both cleaned fresh (NC_01_CL_FLOT) and stored (NC_03_CL_FLOT) waste rock materials were first dried at 40 °C for 12 h, and then dried at 105 °C overnight before incorporation in the facing brick blends.

All four company-specific raw materials (local loam, imported clay A, imported clay B, and imported filler) were dried overnight in a ventilated stove (Heraeus UT 6060) at 105 °C. The dried local loam and dried imported clay A and B, were further processed using a shredding machine (Hosokawa-Alpine MZ-25) in order to break the material and facilitate the integration in the facing brick blends, without changing the natural grain size distribution of the samples (Appendix A).

2.2.2. Lab production of ceramic blends and test specimens

For the design of new facing bricks blends (Table 1B), attention was given to the >50 μm, 50-2 μm, and <2 μm fractions of each new blend, when replacing raw materials with mine waste materials, in order to be in the same range of the standard. The amount of mine tailings incorporated in the facing bricks (20 and 40 wt%) was chosen based on satisfactory results achieved using Plombières tailings material in blends for roof tiles, blocks and hand-moulded pavers in a previous study (Veiga Simão et al., 2021b) and based on the decrease in sulphur and meta (loid)s content of the Neves Corvo waste rock materials after cleaning by froth flotation (Table 1A) when compared to the uncleaned was rock materials (Veiga Simão et al., 2021a).

Preparation of the facing brick test specimens included (1) mixing the dried company specific raw materials with the mining waste materials in a blending machine with the addition of water for good plasticity (Pfefferkorn value of 15 ± 1 mm imprint); (2) moulding the blends by hand using a hand-press with a 110 × 55 × 20 mm mold; (3) marking wet test specimens with a 100 mm caliper mark to measure shrinkage; (4) lab drying (29 °C and 50% RH, overnight) of 15 test specimens per blend; (5) lab firing (electric kiln at 1060 °C, 90 °C/h, 1 h dwell time) (Fig. 1). Additionally, some lab-dried test pieces were fired in an industrial kiln (natural gas kiln at 1060 °C, 49 °C/h, 10 h dwell time) in order to check if lab conditions can simulate industrial conditions.

2.2.3. Physical, mineralogical, chemical, thermal, technical, and aesthetical characterisation

For the physical, mineralogical, and chemical analyses, dried samples were homogenised, grounded with a porcelain mortar and pestle (Morgan Advanced Materials) and sieved at <2 mm, <250 μm, or <200 μm (Retsch sieves), depending on the intended analysis and following internal protocols. For the SEM-EDX analysis of the white staining on the fired body, material from the surface was chipped off.

Specific surface area (SSA) was determined by methylene blue adsorption on powdered samples (<250 μm). The grain size distribution was performed after mixing the samples (<2 mm) with water and sodium tripolyphosphate to de-flocculate the clay fractions for 15 min. The samples were boiled for 10 min and after cooling down the samples were split in two fractions through wet sieving (50 μm). The fraction $>$ 50 μm was dried at 105 °C in a ventilated stove (Heraeus UT 6060) and sieved on a mechanical shaking column with sieves ranging from 1.4 to 0.09 mm (Retsch AS 200 control). The fraction <50 μm was analysed by a sedigraph machine (Micromeritics SediGraph 5100) based on the sedimentation method (Pye and Blott, 2004). Mineralogical characterisation of mining waste materials and fired ceramic samples was performed on powdered samples (<200 μm) by X-ray powder diffraction (XRD) using a Philips Analytical X-ray, model PW1830 generator with a PW3710 mpd control and CuKα radiation at 45 kV and 30 mA. Quantitative identification was based on the Rietveld method (Snellings et al., 2010) using Profex software (version 3.14.3).

Major and trace element concentrations, total carbon and sulphur content, gross loss on ignition (LOI), CO_2 from decomposed carbonates, soluble sulphates and cations, and pH were all determined on powdered samples (<250 μm). Additionally, total concentrations of metal (loid)s and organic compounds were determined on the three mining waste materials (PL_62_I, NC_01_CL_FLOT, and NC_03_CL_FLOT). An overview of the different analytical methods can be found in the supplementary information (Appendix B). The surface of the chips from the white-stained fired test specimens (Appendix C) was analysed by a scanning electron microscope (SEM), using an FEI NOVA NANOSEM 450. For that purpose, samples were coated with Pt–Pd (80 %–20 %). The elemental composition and mapping were performed with an energy dispersive X-ray (EDX) spectrometer (BRUKER QUANTAX 200) equipped with a silicon drift detector (SDD).

Thermal gravimetry and differential scanning calorimetry (TG-DSC) analyses were performed using a Netsch STA 449 F3 machine, by firing the powdered samples (<200 μm) at 1050 °C in a platinum crucible, under oxidising conditions. These thermal analyses were performed on mining waste materials, company-specific raw materials and unfired mixtures to confirm some results of chemical and mineralogical analyses. Nevertheless, results were not reported as these analyses did not bring much more added-value but rather a confirmation of other chemical and mineralogical results.

Moisture content (MC) was determined by the weight difference between wet and dried blends at 105 °C in the ventilated stove (Heraeus UT 6060) and expressed in wt% on a wet basis. The drying behaviour of the hand-moulded wet test specimens were assessed every hour during 8 h for weight loss, drying shrinkage, drying efflorescence, and crack formation using 4 different drying conditions with different temperatures (°C) and relative humidities (RH) (33 °C, 34 % RH; 37 °C, 27 % RH; and 41 °C, 16 % RH) in a lab climate chamber (Vötsch VC3 4060 with S!MPATI® software), and in a ventilated drying stove at 105 °C. The dried test specimens were fired in an electric lab kiln (Fours H&C SPRL, Type 100) at 1060 °C at a speed of 90 °C/h and a dwell time of 1 h. The firing shrinkage and firing colour of each test specimen were compared with the standard. Porosity (water absorption) was deter-

Table 1
Physical, chemical, and mineralogical characterisation of mining waste and company-specific raw materials (A), and composition of facing brick blends on a dry weight basis (B).

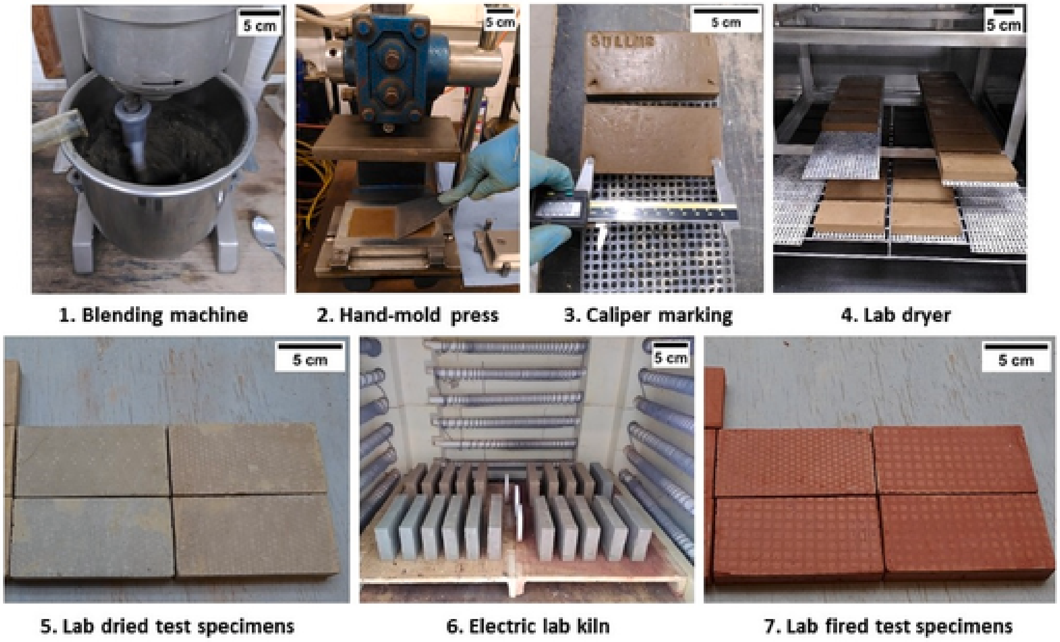
A	Physical, chemical and mineralogical characterisation of mining waste and company-specific raw materials										
Materials	>50 µm	50-2 µm	<2 µm	SSA	$CO_{2carbonates}$	C_{total}	$C_{organic}$	S_{total}	pH		
	wt%	wt%	wt%	m^2/g	wt%	wt%	wt%	wt%			
PL_62_I	14	47	39	93	0.03	0.1	0.1	0.01	7.1		
NC_01_CL_FLOT	6	54	40	23	0.9	0.4	0.2	0.7	6.5		
NC_03_CL_FLOT	17	43	40	26	0.03	0.5	0.5	1.0	4.9		
Local loam	14	47	39	58	0.02	0.2	0.2	<LOD	NM		
Imported clay A	18	24	58	91	0.3	0.1	0.05	0.01	NM		
Imported clay B	23	39	38	70	1.2	0.5	0.2	0.03	NM		
Imported filler	89	NM	NM	12	0.4	0.1	<LOD	<LOD	NM		
Materials	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**TiO_2**	**CaO**	**MgO**	**Na_2O**	**K_2O**	**MnO**	**LOI**	
	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	
PL_62_I	74.0	12.0	4.3	0.9	0.6	0.9	1.1	2.4	0.2	3.3	
NC_01_CL_FLOT	54.9	17.6	12.8	0.7	0.2	2.1	0.3	2.8	0.1	5.8	
NC_03_CL_FLOT	59.7	14.0	11.5	0.6	0.4	1.3	0.6	2.9	<LOD	6.3	
Local loam	80.2	8.1	3.2	0.8	0.6	0.6	1.1	2.0	0.1	2.5	
Imported clay A	73.3	14.3	2.9	1.3	0.3	0.5	<LOD	1.6	<LOD	5.1	
Imported clay B	63.2	17.6	5.5	0.9	0.5	1.1	0.4	3.8	0.2	5.6	
Imported filler	42.0	12.6	11.2	3.0	12.1	9.9	3.3	3.1	0.2	0.6	
Materials	**Ba**	**Cr**	**Cu**	**Ni**	**Pb**	**Zn**	**Soluble SO_4^{2-}**	**Soluble Ca^{2+}**	**Soluble Mg^{2+}**	**Soluble Na^+**	**Soluble K^+**
	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	wt%	mg/kg	mg/kg	mg/kg	mg/kg
PL_62_I	366	60	23	31	30	137	0.01	52	5	13	22
NC_01_CL_FLOT	409	127	402	49	777	1300	0.4	849	505	60	169
NC_03_CL_FLOT	444	91	1181	36	349	462	0.7	2466	182	209	149
Local loam	357	45	14	20	13	42	0.01	176	15	29	32
Imported clay A	212	97	20	41	58	77	0.03	108	33	52	55
Imported clay B	311	96	37	73	24	76	0.04	198	18	73	66
Imported filler	703	140	58	113	<LOD	70	0.03	219	1	94	263
Materials	**Quartz**	**Albite/ Plagioclase**	**Alkali feldspar**	**Pyroxene**	**Kaolinite**	**Chlorite**	**2:1 layer silicates***	**Hematite**	**Rutile**	**Goethite**	**Pyrite**
	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%
PL_62_I	56.1	8.1	5.1	0.5	2.4	3.1	22.1	0.4	0.4	1.1	
NC_01_CL_FLOT	39.2				3.8	19.9	32.4		1.0		1.0
NC_03_CL_FLOT	40.3	2.8			1.8	7.2	27.5	1.1	0.6	2.3	0.6
Materials	**Gypsum**	**Jarosite**	**Siderite**	**Ankerite/ Dolomite**	**Halite**	**Other/ Amorphous**					
	wt%	wt%	wt%	wt%	wt%	wt%					
PL_62_I			0.4	0.2							
NC_01_CL_FLOT	0.1	0.7	1.9								
NC_03_CL_FLOT	0.4	1.5			0.2	13.7					
B	**Composition of facing brick blends on dry weight basis**										
Materials	**LM_1**	**LM_2**	**LM_3**	**LM_4**	**LM_5**	**LM_6**	**LM_7**				
	wt%	wt%	wt%	wt%	wt%	wt%	wt%				
PL_62_I		20.0	40.0								
NC_01_CL_FLOT				20.0	40.0						

(*continued on next page*)

Table 1 (*continued*)

A	Physical, chemical and mineralogical characterisation of mining waste and company-specific raw materials								
Materials	>50 µm	50-2 µm	<2 µm	SSA	$CO_{2carbonates}$	C_{total}	$C_{organic}$	S_{total}	pH
	wt%	wt%	wt%	m^2/g	wt%	wt%	wt%	wt%	
NC_03_CL_FLOT						20.0	40.0		
Local loam	76.0	56.0	36.0	54.0	32.0	56.0	38.0		
Imported clay A	13.5	13.5	13.5	13.5	13.5	13.5	13.5		
Imported clay B	3.5	3.5	3.5	3.5	3.5	3.5	3.5		
Imported filler	7.0	7.0	7.0	9.0	11.0	7.0	5.0		

NM: not measured; LOD: limit of detection; *e.g., Illite/muscovite, smectite, and interstratified Illite/smectite.



1. Blending machine
2. Hand-mold press
3. Caliper marking
4. Lab dryer
5. Lab dried test specimens
6. Electric lab kiln
7. Lab fired test specimens

Fig. 1. Lab production of facing brick test specimens (scale bar: 5 cm).

mined by a 24 h full water immersion test (E_{24h}). The modulus of elasticity (E-modulus) of the fired test specimens was determined by GENEMOD software (version 2.0), based on the specimens' dimensions, weight, and natural vibration (R value). The vibration (µs) was measured by a non-destructive impulse excitation technique based on the ASTM E1876-15 standard (GrindoSonic machine, model MK5 industrial). Finally, an in-house efflorescence sensibility test was performed on the fired test specimens. The specimens were cut in half and placed vertically in approximately 5 mm of distilled water. After 3 days of soaking, the specimens were dried at 50 °C in a ventilated stove (Heraeus UT 6060). This process was repeated three times, and, lastly, the specimens were dried overnight at 105 °C.

2.2.4. *Leaching tests*

Three environmental compliance tests were performed on the standard and mine waste derived fired test specimens with the highest mine waste incorporation (40 wt%) as an optimal valorisation scenario:

- For product application as a shaped building material, a diffusion or tank leaching test (L/A = 80 l/m^2, 8 fractions, 64 days) was performed according to method CMA/2/II/A.9.2 (CMA, 2020) based on NEN 7375:2004 standard on fired test specimens embedded in epoxy resin with only one surface uncovered in order to calculate the emission;
- For second life assessment as a non-shaped building material, a column leaching test (L/S_{cum} = 10 l/kg, 7 fractions, 21 days) was performed on jaw-crushed (Retsch BB 200) and sieved (95 wt% <4 mm) samples according to method CMA/2/II/A.9.1 (CMA, 2020) based on NEN 7373:2004 standard, which included filling up a glass column with pre-treated material up to 4 times the diameter of the glass column (usually 20 cm for 0.5 kg of dried material);
- For evaluation of landfill disposal, a batch leaching test (L/S_{cum} = 10 l/kg) was performed, based on NBN EN12457–2:2002 standard, which included shaking 1 g of powdered sample (<200 µm) with deionised water (10 ml) in a 30 ml Nalgene polypropylene centrifuge tube for 24 h (Edmund Buehler flask shaking machine), before centrifuging (15 min, 3200–3500 rpm, Beckman GS-6 centrifuge). Normally, the batch leaching test is carried out on crushed and sieved material (<4 mm); therefore, the leaching of powdered samples (<200 µm) represents a worst-case scenario.

Element concentrations in the acidified leachates (addition of 5 wt% of HNO_3 at 65 %) from the column and diffusion leaching tests were determined by ICP-OES (Agilent Technologies 5100), according to method CMA/2/I/B.1 (CMA, 2020). For the batch leaching test, element concentrations were determined in acidified leachates (addition of one drop of HNO_3 at 65 %) with ICP-OES (Varian 730 ES).

2.2.5. Life cycle assessment

The environmental performance of standard facing bricks (LM_1) and waste-derived facing bricks made with 40 wt% Plombières tailings (LM_3) was assessed by LCA following the ISO 14040:2006 standard. The latter brick was selected as an optimal valorisation scenario, and because Plombières tailings material is the only viable alternative material in terms of quality and sourcing feasibility. Based on the current investigation, facing bricks containing 40 wt% of Plombières tailings show satisfactory technical and aesthetical properties and comply with environmental requirements (leaching of metal (loid)s). The functional unit for the LCA is defined on a product mass basis as “1 tonne of ceramic bricks”. Transport of raw materials from suppliers and from the mine tailings site to the brick factory was included in the respective subsystems. The installation, use phase, and end-of-life were excluded in the analysis because both products achieve the same technical specifications and same utilisation until reaching the disposal and end-of-life phase.

Concerning *system boundary and scenarios*, the LCA has followed a cradle-to-gate approach, considering the following life cycle phases (Fig. 2): Raw materials acquisition, transport, and manufacturing.

For the *life cycle inventory analysis*, the main sources of foreground inventory data were gathered from the latest company's technical report. Exchanges with the representatives were conducted to collect detailed materials, energy, and emissions input and output data of a specific brick manufacturing plant in Belgium. The background inventory data were obtained from the Ecoinvent 3.6 database (Ecoinvent, 2019). The life cycle models were constructed and simulated using SimaPro 9.2 software.

The product environmental footprint (PEF) version 3.0 method was used to evaluate the environmental impacts of ceramics production in the *life cycle impact assessment phase*. This method includes 16 midpoint indicators across impact categories and a single score endpoint indicator, using weighting factors (Fazio et al., 2018). Also, the impact assessment method is in line with the harmonized European standard for product declaration (Product Environmental Footprint Category Rules). This allows for the consistent application of midpoint and endpoint methods (European Commission, 2017).

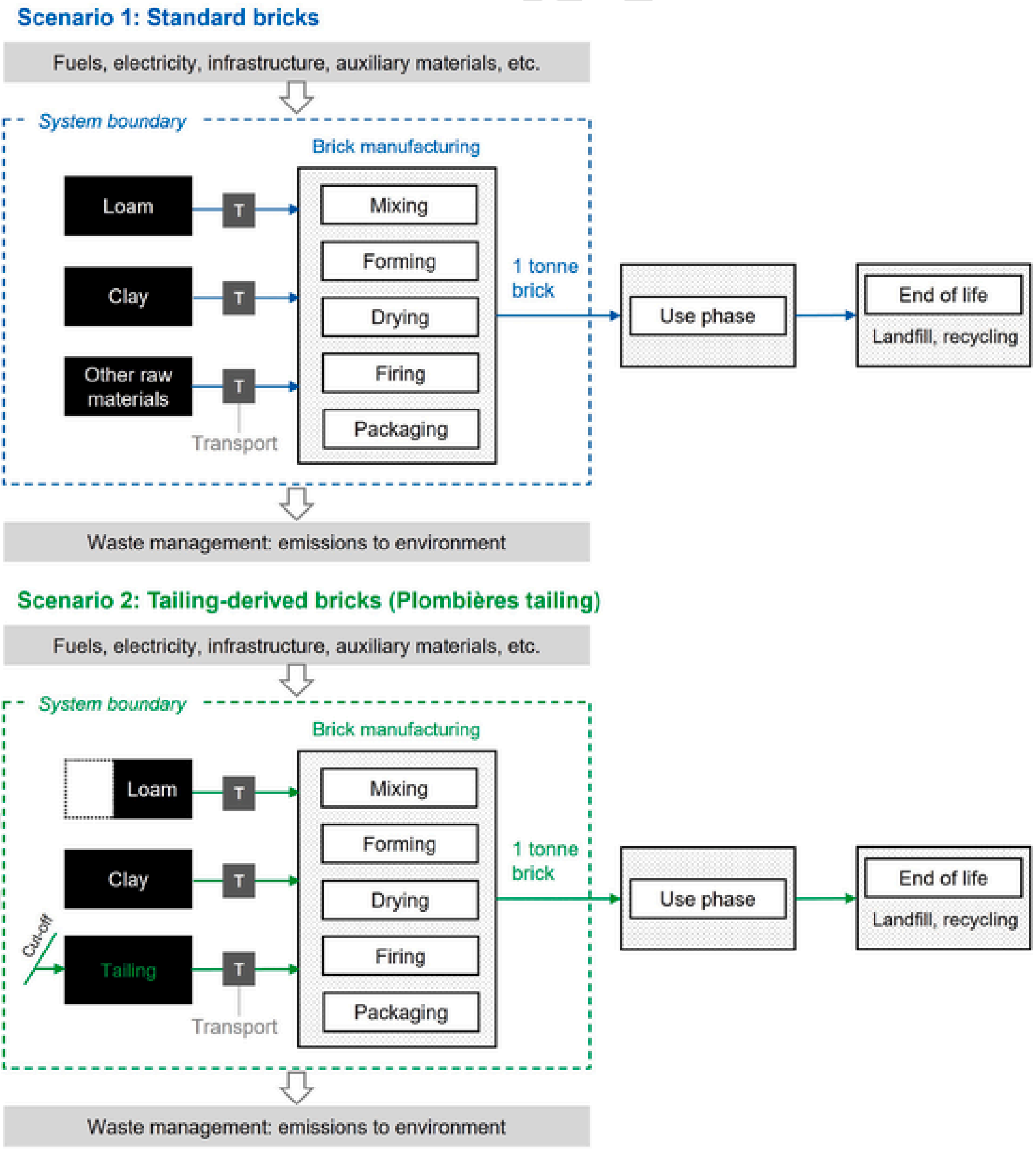


Fig. 2. Life cycle flow chart of standard bricks (Scenario-1) and tailings-derived bricks (Scenario-2).

3. Results

3.1. Materials characterisation

3.1.1. Mining waste

The Plombières tailings material (PL_62_I) is a fine-grained material (86 wt% <50 µm) with a specific surface area (SSA) of 93 m^2/g (Veiga Simão et al., 2021a). This tailings material has a neutral pH (Table 1A) and mainly contains silicates (quartz (SiO_2), feldspar (e.g., plagioclase $NaAlSi_3O_8$ – $CaAl_2Si_2O_8$), 2:1 layer silicates (e.g., muscovite $KAl_2(AlSi_3O_{10})(OH)_2$), and pyroxene ($AD[Si_2O_6]$), some oxides (goethite (α-FeO(OH)), hematite (Fe_2O_3), rutile (TiO_2)), and carbonates (siderite ($FeCO_3$) and dolomite ($CaMg(CO_3)_2$) or ankerite ($Ca(Fe^{2+},Mg)(CO_3)_2$)) (Table 1A and Appendix D). The major elemental composition (XRF) is in line with the mineralogical composition, with silicium (74.0 wt% SiO_2) and aluminium (12.0 wt% Al_2O_3) being the main elements (Table 1A). The loss on ignition (LOI) is low (3.3 wt%) compared to the other mining waste and clay materials but similar to the LOI of local loam (Table 1A). The total sulphur content of the Plombières tailings material is very low (0.01 wt% S), and, consequently, also the soluble sulphate content (0.01 wt% SO_4^{2-}) (Veiga Simão et al., 2021a).

The cleaned Neves Corvo fresh waste rock material (NC_01_CL_FLOT) shows a very fine grain size (94 wt% <50 µm) as a result of the pre-treatment, and a relatively low SSA (23 m^2/g) (Table 1A and Appendix A). This neutral waste rock material (pH = 6.5) is mainly composed of silicates (quartz and 2:1 layer silicates) and chlorite ($(Mg,Fe)_3(Si,Al)_4O_{10}(OH)_2 \cdot (Mg,Fe)_3(OH)_6$)), some oxides (rutile), sulphides (pyrite (FeS_2), sulphates (jarosite ($KFe_3(SO_4)_2(OH)_6$) and gypsum ($CaSO_4 \cdot 2H_2O$)), and carbonates (siderite) (Table 1A and Appendix E). The major elemental composition shows a correlation with the mineralogical composition with silicium (54.9 wt% SiO_2), aluminium (17.6 wt% Al_2O_3), and iron (12.8 wt% Fe_2O_3) being the most abundant elements (Table 1A). The LOI value is high (5.8 wt%) when compared to Plombières tailings material. This high LOI can be explained by the oxidation and release of SO_x (pyrite), CO_2 (organic C), the decomposition of siderite with release of CO_2, and the release of water and SO_x from jarosite and gypsum. Soluble sulphate (0.4 wt% SO_4^{2-}), Ca^{2+} (849 mg/kg), Mg^{2+} (505 mg/kg), and K^+ (169 mg/kg) contents are high when compared to the other materials, except for the clean Neves Corvo stored waste rock where higher values are in line with the jarosite and gypsum concentrations (Table 1A).

The cleaned Neves Corvo stored waste rock (NC_03_CL_FLOT), just like NC_01_CL_FLOT, is a fine-grained material (83 wt% <50 µm) with a low SSA (26 m^2/g) (Table 1A and Appendix A). This acidic waste rock material (pH = 4.9), is composed of silicates (quartz, feldspar, and 2:1 layer silicates), some oxides (goethite, hematite, rutile), sulphides (pyrite), sulphates (jarosite and gypsum), traces of halides (halite (NaCl)), as well as a considerable amount of amorphous material (Table 1A and Appendix F). The amorphous material detected in this sample could be related to the origin of the waste rock that is stored in an temporary open-air waste rock pile with visible weathered layers and common minerals that are a result of weathering (e.g., halite). Additionally, cleaning by froth flotation changes the chemical stability of mineral phases, and may also generate amorphous phases. Major element composition is in line with mineralogical results and includes silicium (59.7 wt% SiO_2), aluminium (14.0 wt% Al_2O_3), and iron (11.5 wt% Fe_2O_3) as the most abundant elements (Table 1A). The high LOI (6.3 wt%) is also linked to the high organic carbon content (0.5 wt% $C_{organic}$), the presence of pyrite (SO_x release), and minerals such as goethite, gypsum, and jarosite (Table 1A). The soluble sulphate content is high (0.7 wt% SO_4^{2-}), as well as soluble Ca^{2+} (2466 mg/kg), Mg^{2+} (182 mg/kg), Na^+ (209 mg/kg), and K^+ (149 mg/kg), and is linked to the jarosite and gypsum concentrations (Table 1A).

3.1.2. COMPANY-SPECIFIC raw materials

Concerning the company-specific raw materials (Table 1A), the local loam is a fine-grained material (86 wt% <50 µm) similar to the Plombières tailings material but with a lower SSA (58 m^2/g). This local material is poor in carbon (0.2 wt% total C) and soluble sulphates (0.01 wt% SO_4^{2-}) but, like many loams, rich in silicium (80.2 wt% SiO_2). The local loam shows a negligible content of metal (loid)s (Cu, Ni, Pb, and Zn). The imported clay A is a fine-grained material (82 wt% <50 µm) with a moderate SSA (91 m^2/g). The total carbon (0.1 wt%), total sulphur (0.01 wt%), as well as the content of soluble sulphates (0.03 wt%) and Ca^{2+} (108 mg/kg) is low when compared to the other materials (Table 1A). The other imported clay (B) is slightly coarser (77 wt% <50 µm) than the other clay with a lower SSA (70 m^2/g) and a high carbonate content (1.2 wt% $CO_{2\ carbonates}$), probably from dolomite, while the organic carbon (0.2 wt%), total sulphur (0.03 wt%), and soluble sulphates (0.04 wt%) content are low (Table 1A). Compared to the other company-specific materials, the imported clay B has the highest LOI (5.6 wt%) mainly due to high carbonate content. However, the high aluminium content and moderate SSA predict the presence of kaolinite (Table 1A). The imported filler is much coarser than the clays (89 wt% >50 µm) and has, by consequence, a very low SSA (12 m^2/g). The LOI is very low (0.6 wt%), as well as the total sulphur and carbon content (Table 1A). Concentrations of minor elements, such as Ba (703 mg/kg), Cr (140 mg/kg), and Ni (113 mg/kg), are higher compared to the other materials. The soluble sulphate content is in range with the other materials (0.03 wt% SO_4^{2-}) but the content in soluble K^+ (263 mg/kg), Ca^{2+} (219 mg/kg), and Na^+ (94 mg/kg) is more pronounced (Table 1A).

3.1.3. Unfired blends

The Plombières tailings-containing blends (LM_2 and LM_3) and Neves Corvo fresh (LM_4–5) and stored (LM_6–7) waste rock-containing blends show the same fine grain size distribution (81 wt% <50 µm) as the standard blend (LM_1) (Table 2). The two blends with Plombières tailings material (LM_2–3) show physical and chemical properties that are comparable with the standard (LM_1) (Table 2), as the Plombières tailings material and the local loam (which is partly replaced by the tailings material), have very similar properties (Table 1A). In contrast, all blends with cleaned Neves Corvo waste rock materials (LM_4–7) have higher contents of total S, soluble sulphates, and soluble cations (Ca^{2+}, Mg^{2+}, Na^+, K^+) because of the more sulphidic nature of the waste rock materials when compared to the partly replaced local loam and imported filler. The blends with cleaned Neves Corvo fresh waste rock (LM_4–5) contain more carbonates (Table 2) coming from siderite in the waste rock sample (NC_01_CL_FLOT) – Table 1A. A slight increase of organic C in the blends with cleaned Neves Corvo stored waste rock (LM_6–7) was expected due to the high content in organic C in the waste rock sample (NC_03_CL_FLOT) – Table 1A. As expected, all the blends with cleaned Neves Corvo waste rock materials, show a lower silicium content but higher amounts of aluminium, iron, and potassium as major elements and Cu, Ni, Zn, and Pb as minor elements (Table 2). After drying, efflorescence marks (bassanite formation) were visible on the surface of the dried test specimens (Fig. 1).

3.1.4. Fired blends

Concerning the lab-fired blends (1060 °C), there is a clear difference between the standard, Plombières tailings, and the cleaned Neves Corvo waste rock blends (Table 3A). The blends with the cleaned Neves Corvo waste rock (LM_4–7) contain high total sulphur content, which increases with the amount of waste that is added, and have at least 10 times more soluble sulphates (SO_4^{2-}) and soluble cations (Ca^{2+}, Mg^{2+}, Na^+, K^+) than the standard or Plombières tailings blends (Table 3A).

As expected, when compared to the corresponding unfired blends, the content of soluble sulphates and soluble cations was considerably decreased by firing in a lab kiln at high temperatures (1060 °C). After

Table 2
Physical and chemical characterisation of unfired facing brick blends.

Unfired blends	>50 µm	50-2 µm	<2 µm	SSA	$CO_{2\ carbonates}$	C_{total}	$C_{organic}$	S_{total}
	wt%	wt%	wt%	m^2/g	wt%	wt%	wt%	wt%
LM_1	19	38	43	74	0.1	0.2	0.2	0.02
LM_2	19	37	44	86	0.1	0.2	0.2	0.02
LM_3	19	37	44	84	0.1	0.2	0.2	0.02
LM_4	19	41	40	71	0.3	0.3	0.2	0.1
LM_5	19	38	43	70	0.5	0.4	0.2	0.3
LM_6	20	37	43	71	0.1	0.3	0.3	0.2
LM_7	19	37	44	70	0.1	0.4	0.3	0.3

Unfired blends	**SiO_2**	**Al_2O_3**	**Fe_2O_3**	**TiO_2**	**CaO**	**MgO**	**Na_2O**	**K_2O**	**MnO**	**LOI**
	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%
LM_1	76.3	9.8	3.9	1.0	1.1	1.2	0.9	2.3	0.1	3.0
LM_2	75.3	10.7	4.1	0.9	1.1	0.9	0.9	2.4	0.1	3.2
LM_3	74.4	10.8	4.3	0.9	1.3	1.1	1.0	2.3	0.1	3.3
LM_4	69.9	11.6	6.4	1.0	1.4	1.6	0.9	2.6	0.1	3.6
LM_5	64.0	13.6	8.4	1.0	1.5	2.0	0.8	2.7	0.1	4.4
LM_6	71.4	11.4	5.6	1.0	1.2	1.2	0.9	2.4	0.1	3.8
LM_7	67.0	12.9	7.5	0.7	1.0	1.2	0.8	2.7	<LOD	4.7

Unfired blends	**Ba**	**Cr**	**Cu**	**Ni**	**Pb**	**Zn**	**Soluble SO_4^{2-}**	**Soluble Ca^{2+}**	**Soluble Mg^{2+}**	**Soluble Na^+**	**Soluble K^+**
	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	mg/kg	wt%	mg/kg	mg/kg	mg/kg	mg/kg
LM_1	373	74	21	36	19	49	0.01	130	23	89	80
LM_2	370	68	21	32	16	65	0.01	213	28	91	83
LM_3	376	70	25	35	18	71	0.01	207	30	85	107
LM_4	406	87	97	45	169	302	0.05	174	26	113	91
LM_5	388	103	169	47	344	599	0.2	453	92	146	172
LM_6	373	75	247	37	83	145	0.2	629	70	145	111
LM_7	390	83	480	38	150	233	0.4	1199	188	206	191

Table 3
Chemical and mineralogical characterisation (A) and technical properties (B) of ceramic test specimens.

A — Chemical and mineralogical characterisation of lab fired test specimens

Lab fired 1060°C	**C_{total}**	**S_{total}**	**Soluble SO_4^{2-}**	**Soluble Ca^{2+}**	**Soluble Mg^{2+}**	**Soluble Na^+**	**Soluble K^+**
	wt%	wt%	wt%	mg/kg	mg/kg	mg/kg	mg/kg
LM_1	0.02	0.01	0.001	22	2	7	5
LM_2	0.02	0.02	0.001	8	1	8	5
LM_3	0.02	0.02	0.002	9	1	9	7
LM_4	0.02	0.02	0.01	20	3	11	10
LM_5	0.02	0.03	0.01	45	5	14	14
LM_6	0.02	0.03	0.01	63	4	11	9
LM_7	0.02	0.04	0.03	134	5	11	11

Lab fired 1060°C	**Quartz**	**Albite/Plagioclase**	**Mullite**	**Pyroxene**	**Alkali feldspar**	**Hematite**	**Spinel-type minerals**	**Other/ Amorphous**
	wt%	wt%	wt%	wt%	wt%	wt%	wt%	wt%
LM_1	56.8	4.6	2.9	2.0	1.1	2.8	0.8	29.0
LM_2	56.5	5.0	3.3	2.3	2.0	3.4	0.8	26.7
LM_3	60.2	5.2	3.4	2.0	1.1	3.7	0.7	23.6
LM_4	57.1	5.4	5.0	2.6	1.8	5.0	1.6	21.5
LM_5	42.4	4.6	7.9	4.4	1.5	6.6	2.6	29.9
LM_6	57.1	3.5	4.9	2.8	1.5	5.1	1.3	23.9
LM_7	51.7	3.5	7.5	1.7	2.4	6.8	1.6	24.8

B — **Technical properties of ceramic test specimens**

Ceramic test specimens	**Moisture content**[a]	**Drying shrinkage**	**Firing shrinkage**		**Water absorption (E_{24})**		**E-modulus strength**	
	wt%	%	%	%	wt%	wt%	GPa	GPa
	Wet blend	**Lab dryer (29°C, 50% RH)**	**Lab kiln (1060°C)**	**Ind kiln (1060°C)**	**Lab kiln (1060°C)**	**Ind kiln (1060°C)**	**Lab kiln (1060°C)**	**Ind kiln (1060°C)**
LM_1	16.0 ± 0.1	4.9 ± 0.6	1.6 ± 0.3	1.2 ± 0.1	8.1 ± 0.3	8.2 ± 0.1	11.2 ± 0.6	10.4 ± 1.1
LM_2	15.9 ± 0.1	5.2 ± 0.5	1.8 ± 0.2	1.8 ± 0.2	7.7 ± 0.01	7.4 ± 0.2	13.2 ± 0.7	12.2 ± 1.1
LM_3	16.6 ± 0.1	5.0 ± 0.3	2.3 ± 0.1	2.4 ± 0.2	6.8 ± 0.005	6.4 ± 0.03	15.2 ± 0.6	15.9 ± 0.5
LM_4	15.3 ± 0.2	4.5 ± 0.6	2.4 ± 0.2	2.4 ± 0.1	6.5 ± 0.2	6.7 ± 0.1	18.4 ± 0.6	17.5 ± 0.5
LM_5	15.4 ± 0.2	4.2 ± 0.3	4.1 ± 0.1	4.3 ± 0.1	4.9 ± 0.3	4.8 ± 0.1	28.2 ± 0.9	27.7 ± 0.5
LM_6	15.7 ± 0.01	4.5 ± 0.5	2.3 ± 0.2	2.6 ± 0.2	7.0 ± 0.1	6.8 ± 0.3	17.0 ± 1.0	15.8 ± 0.2
LM_7	15.8 ± 0.1	4.4 ± 0.2	3.6 ± 0.1	3.9 ± 0.1	6.1 ± 0.01	5.8 ± 0.1	21.8 ± 0.6	22.3 ± 0.04

[a] On wet basis and for a Pfefferkorn value of 15 ± 1 mm imprint; RH: Relative Humidity; Lab: Laboratorial; Ind: Industrial.

firing, white-staining marks appeared on the surface of the test specimens using cleaned Neves Corvo waste rock materials (Fig. 1), making the firing colour of these test specimens visually different than the standard with a naked eye. XRD results of the white-staining marks were not conclusive but a SEM-EDX analysis showed a high content of Al and Ca, and lower Si concentrations when compared to non-staining areas (Appendix C). XRD results of the fired blends show presence of silicates (quartz, feldspar, mullite ($Al_{4+2x}Si_{2-2x}O_{10-x}$), pyroxene), oxides (hematite and spinel-type minerals ($MgAl_2O_4$)), as well as a considerable amount of amorphous phase (Table 3A). The amounts of the different mineral phases slightly differ from blend to blend (Table 3A). The diffractograms with the indication of the main mineral phases of the fired brick blends can be found in Appendix G.

3.2. Technical, aesthetical, and chemical properties of test specimens

The climate chamber test showed a similar to slightly better drying behaviour (speed of weight loss and appearance of cracks) for the test specimens with Plombières tailings (LM_2–3) and with cleaned Neves Corvo fresh waste rock (LM_4–5), when compared to the standard (LM_1). Cleaned Neves Corvo stored waste rock test specimens (LM_6–7) showed a less good drying behaviour. After drying, efflorescence was visible in all lab test specimens containing cleaned Neves Corvo waste rock materials. Concerning moisture content and drying shrinkage results, only slight differences were found between the standard and the mine waste-containing test specimens (Table 3B).

The dried test specimens were fired in a lab kiln and in an industrial kiln (1060 °C). There were no significant differences in technical properties between the lab and industrial fired test specimens, meaning that the lab kiln simulates very well the industrial kiln. The test specimens with Plombières tailings material (LM_2–3) show slightly better properties (higher firing shrinkage, lower water absorption, and higher E-modulus strength) than the standard test specimens (LM_1) (Table 3B). Therefore, Plombières tailings material can be considered a good alternative for the local loam. The blends containing the cleaned Neves Corvo waste rock materials show a further increase of the firing shrinkage and E-modulus strength and, consequently, a further decrease of water absorption (Table 3B). This pattern is more pronounced in the test specimens with 40 wt% mine waste incorporation (Table 3B). The better technical properties of the test specimens using cleaned Neves Corvo waste rock material can be linked to the replacement of local loam, poor in fluxing agents, by waste rock materials with higher amounts of fluxing agents, such as iron (Fe_2O_3), magnesium (MgO), and potassium (K_2O) (Table 2), which generally promotes better technical performances of fired bricks (Rehman et al., 2020). Moreover, the high mullite content in fired blends with cleaned Neves Corvo waste rock (Table 3A) explains the higher E-modulus strength (Table 3B) (Schneider et al., 2015).

The efflorescence on the dried test specimens containing cleaned Neves Corvo waste rock becomes a white-staining on the fired test specimens (Fig. 1 and Appendix C). After a mineralogical analysis of the white dry efflorescence, it was possible to identify bassanite ($CaSO_4 \cdot \frac{1}{2} H_2O$). The highly soluble SO_4^{2-} and Ca^{2+} content in these unfired blends (Table 2) reach the drying surface through capillarity-flow resulting in efflorescence (Eloukabi et al., 2013). However, the mineralogical analysis of the white staining marks on the surface of fired test specimens did not show a conclusive mineral phase, but an SEM-EDX analysis showed the presence of Ca, Al, and Si (Appendix C). Concerning the firing colour, only the test specimens containing Plombières tailings material show the same surface and body (Fig. 3) colour as the standard.

The efflorescence test, performed on the fired test specimens, showed efflorescence formation only on the test specimens with cleaned Neves Corvo waste rock materials (Fig. 3), especially when they contained stored waste rock (LM_6–7), which has the most soluble SO_4^{2-} and Ca^{2+} content (Table 3A). The XRD analysis of the white salt formation (efflorescence) identified a hemihydrate form of calcium sulphate (bassanite).

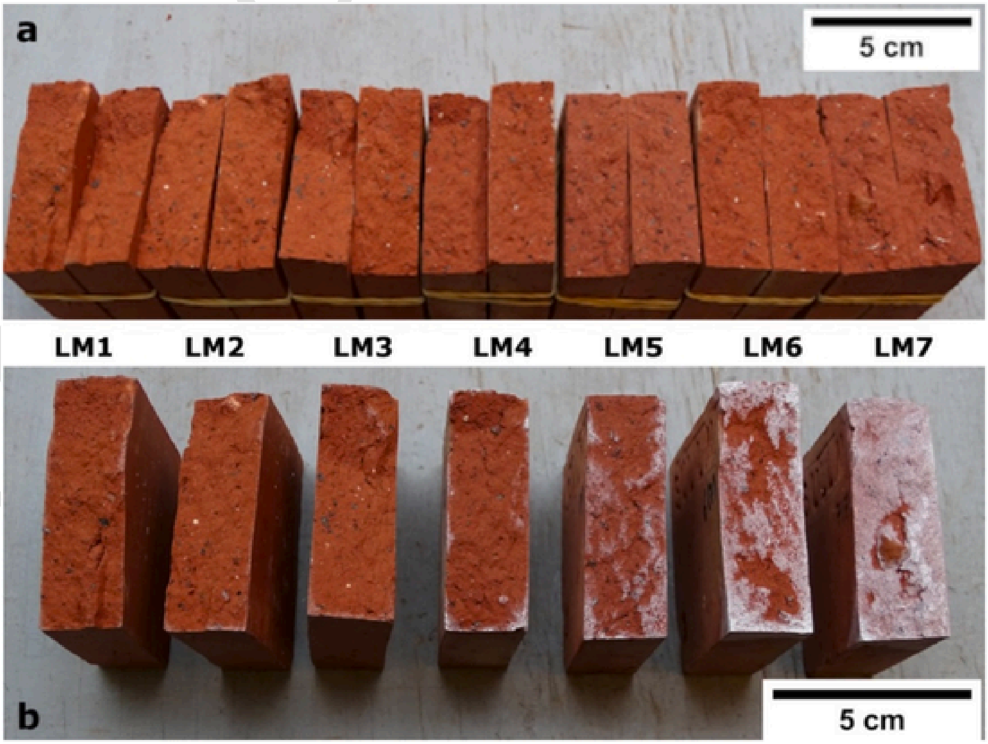


Fig. 3. Aesthetical properties of lab-fired facing brick test specimens (1060 °C): (a) intersection; (b) efflorescence test result.

3.3. Environmental compliance tests on fired test specimens

The diffusion, column, and batch leaching tests were performed on the lab-fired (1060 °C) standard test specimen (LM_1) and test specimens with 40 wt% mining waste incorporation (LM_3, 5, and 7).

In a *first life scenario* (service life/use phase), the results from the diffusion test show that both of the fired bricks with 40 wt% cleaned Neves Corvo waste rock (LM_5 and LM_7) exceed the limit values for arsenic (As) leaching, meaning that these materials cannot be used in shaped building materials (VLAREMA, 2012) (Appendix H).

In a *second life scenario*, the results from the column test also show that both fired bricks with cleaned Neves Corvo waste rock (LM_5 and 7) exceed the limit values for As leaching (VLAREMA, 2012), especially LM_7 with stored waste rock (Appendix H). Considering the failure in the diffusion test, this result is not surprising. As a result, cleaned Neves Corvo fresh or stored waste rock materials cannot be used in shaped building materials, such as facing bricks, at least with 40 wt% incorporation.

The standard (LM_1) and the Plombières-tailings (LM_3) bricks were within regulatory limits in both scenarios (Appendix H) and can be either produced as shaped building materials (first life) or used in a second life scenario where shaped building materials are demolished and recycled/downcycled as non-shaped building materials (e.g., aggregates). For the assessment of pH-dependency of metal (loid)s leaching during the column test, results show that As leaching is pH-dependent for the standard (LM_1) and the tailings-containing (LM_3) test specimens (R^2 = 0.8 and 0.5, respectively) (Fig. 4). The pH-dependent leaching of As in the fired test specimens shows that As is more mobile as pH rises. The first eluate fraction was not considered due to its influence by wash-off (Fig. 4).

In an *end-of-life scenario*, the results from the batch leaching test show that the standard (LM_1) and Plombières tailings-containing (LM_3) test specimens are under the limit values and can be potentially landfilled as inert waste (European Commission, 2003). On the contrary, both cleaned Neves Corvo waste rock-containing test specimens (LM_5 and 7) exceed the limit values for inert and for non-hazardous waste (excessive leaching of As), and can only be landfilled as hazardous waste (European Commission, 2003) (Appendix H).

The leaching concentrations of most elements remained below regulatory limits in the different eluate fractions of the diffusion and column leaching tests. This is the case for Cd, Cr, Cu, Pb, Ni, and Zn leached from the standard (LM_1) and Plombières tailings-containing (LM_3) test specimens, and for Cd, Pb, and Ni leached from both Neves Corvo waste rock-containing test specimens (LM_5 and 7). In the diffusion

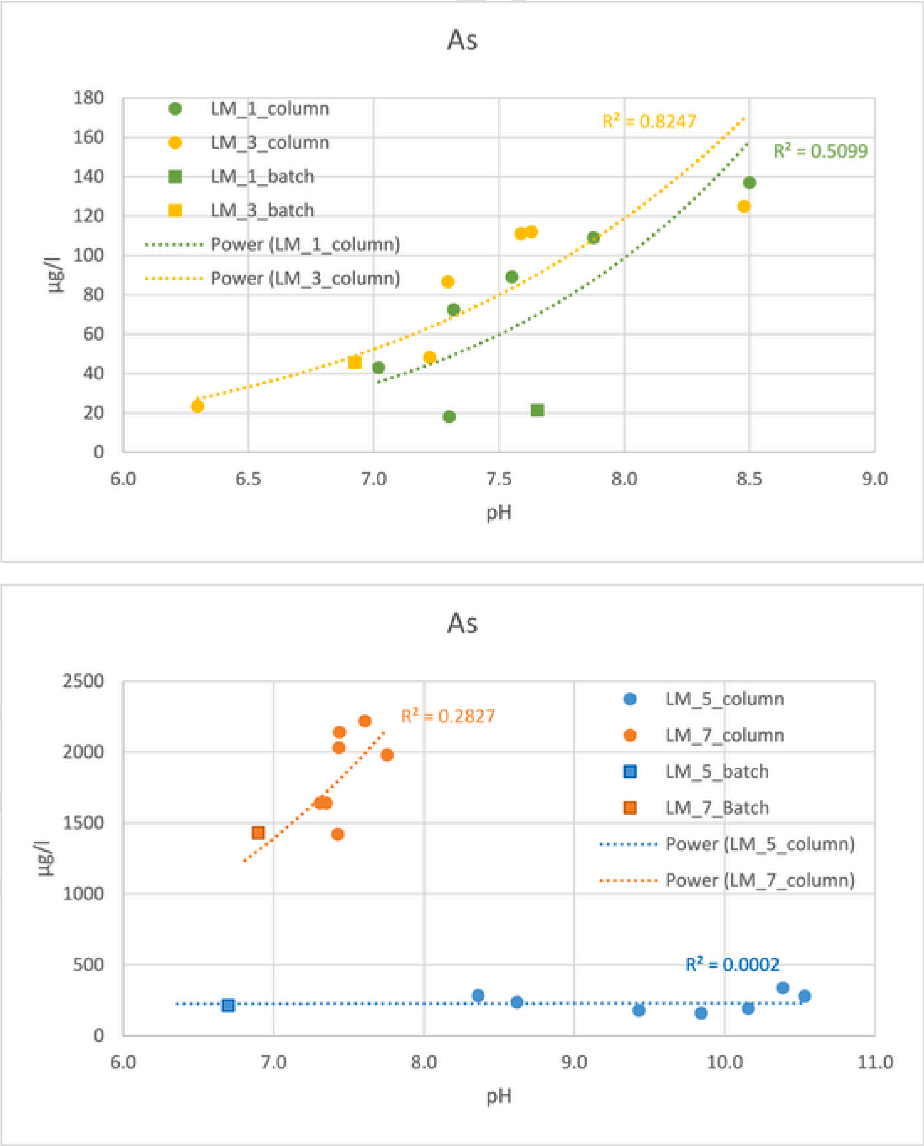


Fig. 4. Leaching of As from fired facing brick test specimens during column and batch leaching tests.

test, As leaching showed measurable concentrations in all eluate fractions for LM_5 and 7, and in the last 4 eluate fractions of LM_1 and 3. The As leaching was, in all cases, diffusion-controlled. In the column test, As showed measurable concentrations in all eluate fractions of all test specimens. In both LM_5 and 7, the leaching of As showed similar concentrations in all 7 eluate fractions, indicating that As leaching is controlled by solubility. This is in agreement with the results of the batch leaching test, which showed similar As leaching for LM_5 and 7, although the material was ground much finer (<200 μm compared to < 4 mm for the column leaching test). The observed difference in leaching results between the batch and column leaching tests of LM_5 can be attributed to the difference in pH between the two leaching tests (Appendix H), as the leaching was shown to be pH-dependent (Fig. 4). The different leaching concentrations in the eluates of LM_5 (160–280 μg/l) and LM_7 (1400–2200 μg/l) during the column and batch leaching tests (Fig. 4) indicate that it is not the same phase that is controlling the leaching concentrations in these two Neves Corvo waste rock containing test specimens.

3.4. *Life cycle assessment of standard and tailings-containing facing bricks*

3.4.1. *Contribution analysis*

The contribution analyses for the two selected facing bricks (LM_1 and 3) showed that energy, direct process emissions in the manufacturing stage, and raw materials are primarily responsible for the dominant environmental impacts in multiple impact indicators (Appendix I). The extraction of natural resources, such as clay, loam, and filler materials, have the highest contribution to health impacts due to fine particulate matter emissions, human and eco-toxicity, eutrophication, and dominant resource depletion impacts for standard bricks. Producing bricks with alternative materials (scenario 2) reduces the ecological pressures in the raw materials stage at varying levels (from 1 % up to 15 % reduction), with the resource use-, toxicity-, and eutrophication-related impact categories demonstrating the largest benefits from such alternative sourcing strategy. Replacing loam with tailings clearly avoids primary mining and extraction processes, which have large environmental impacts on resource depletion and pollutant releases. While resources can be saved through this alternative scenario, burden shifts still occur due to additional impacts induced by the transport of tailings materials to the brick manufacturing plant.

3.4.2. *Comparative evaluation*

A side-by-side environmental profile of conventional bricks (scenario 1) and waste-derived bricks (scenario 2) illustrates that the latter has varying benefits across impact indicators (Fig. 5). Although energy- and fossil-related impact categories (e.g., climate change, resource use, fossils) show less than 1 % benefits in scenario 2, prominent advantages are attributed to toxicity and mineral depletion impact categories. Scenario 2 can reduce the impacts by more than 10 % for particulate matter, human and eco-toxicity, eutrophication, and even by one-third for minerals and metals resource use impacts. No significant improvements are observed for climate change impacts, while the single score results can be reduced by 7 %. In sum, the environmental gains from reusing waste can offset additional burdens from transport. Overall, this study highlights that alternative sourcing, particularly for producing fired bricks that comply with market standards regarding production processes, product quality, and environmental performance, also has environmental benefits from a life cycle perspective.

4. Discussion

4.1. *Feasibility of using mine waste in facing bricks*

The facing brick blends with Plombières tailings material (LM_2–3) have technical, aesthetical, and chemical properties very similar to the standard blend (LM_1). There is no significant difference in drying behaviour (weight loss, drying shrinkage, crack formation), and the test specimens are, like the standard, free from drying efflorescence. The fired specimens performed slightly better than the standard, with higher firing shrinkage, E-modulus strength, and lower porosity (water absorption), but the difference was only significant for the samples with higher incorporation of cleaned Neves Corvo fresh (LM_5) and stored (LM_7) waste rock materials (Table 3B). No efflorescence was visible on the fired test specimens with Plombières tailings material (LM_2–3) (Fig. 3b), mainly due to their low concentrations of soluble SO_4^{2-} and Ca^{2+} (Table 3A). Moreover, the firing colour of the lab test specimens was in the same range as the standard (Fig. 3a). Therefore, Plombières tailings material is a good alternative for the local loam.

Although the facing brick blends using cleaned Neves Corvo waste rock materials (LM_4–7) showed satisfactory firing properties compared with the standard (LM_1) (Table 3B), the high content of highly

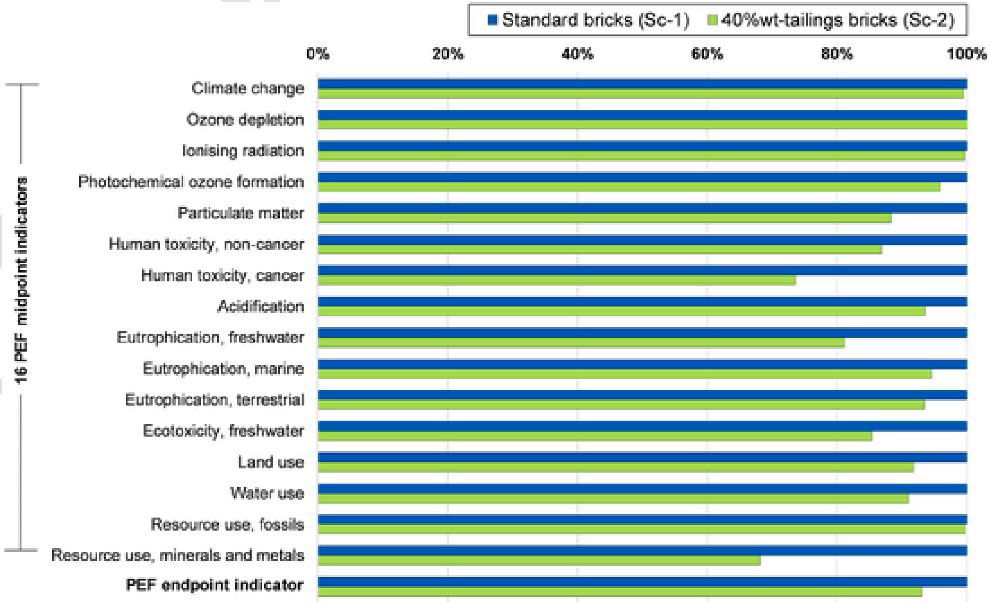


Fig. 5. Comparative environmental impact values of standard bricks (Sc-1) and tailings-derived bricks (Sc-2). The environmental impact of the standard is always set at 100 %.

soluble SO_4^{2-} and Ca^{2+} together with pore moist flow (Janssen et al., 2020) promoted a strong efflorescence (bassanite formation, as confirmed by XRD) on the test specimens after drying (Fig. 3). When dissolved in water, the highly soluble ions (SO_4^{2-} and Ca^{2+}) can reach the surface of porous materials, such as ceramic bricks, by diffusion (ions movement within the water driven by salt concentration) and advection (transport of water carrying the salt ions) (Chwast et al., 2015).

Moreover, after firing, white-staining marks were visible on the waste rock containing test specimens. Due to the thin layer of white-staining, it was not possible to identify a clear mineral phase by XRD. An SEM-EDX analysis identified presence of high Ca and Al concentrations, and lower Si concentrations when compared to the non-staining areas on the surface of the fired test specimens (Appendix C).

In Flanders (Belgium), a waste material complying with the regulatory leaching limits (column leaching test) and total concentrations of organic compounds (VLAREMA, 2012) can obtain a raw material declaration. The material is then no longer considered a waste (end-of-waste status), and can be used by the industry, as non-shaped building material, in the same way as any other primary raw material. This raw material declaration is important since it facilitates the reuse of 'waste materials' in building and construction applications. The Plombières tailings material (PL_62_I) proved to be suitable for use as non-shaped building material (e.g., cover layers, rock-earth filled dams) since it complied with regulatory limit values for organic compounds and metal (loid)s leaching (Veiga Simão et al., 2021a).

Moreover, if the (waste) material is intended to be used in or as a shaped building product, then an additional environmental compliance with the regulatory limits for metal (loid)s leaching during service life (diffusion leaching test) is mandatory (VLAREMA, 2012).

The Plombières tailings-containing (40 wt%) facing bricks showed a leachability of metal (loid)s below the limit values for the diffusion leaching test (Appendix H), meaning that it is feasible to use this tailings material as a raw material (40 wt%) for facing brick blends. The facing brick blends with the highest incorporation of the cleaned Neves Corvo waste rock materials (LM_5 and 7), especially the stored waste rock material (LM_7), showed leachability of As above limit values during the regulatory test (diffusion test) for service life (Appendix H), which means that the cleaned Neves Corvo waste rock materials cannot be used as alternative raw materials in that quantity (40 wt%) for this company-specific facing brick blend. This suggests that both cleaned Neves Corvo waste rock materials should be further cleaned from sulphur and metal (loid)s before incorporation in ceramics, preferably using low-impact cleaning techniques for recovering valuable base and critical metals (Rodriguez et al., 2021). This high sulphur and metal (loid)s content of sulphidic mine waste materials can be considered as a main challenge as the immobilisation of hazardous metal (loid)s tends to be difficult when sulphide and/or sulphate minerals are abundant in building materials (Martins et al., 2021).

4.2. *Environmental impact of using mine waste as a raw material for facing bricks*

The continuous efforts on repurposing waste from other industries as alternative materials in fired bricks manufacturing are proven to be promising. Several studies on the LCA of waste-based fired bricks from underutilised streams stress the importance of alternative sources: slag (Muñoz et al., 2018), fly ash (Huang et al., 2017), and bio waste (Bories et al., 2016). The calculated environmental benefits generally showed that incorporating specific amounts of waste in bricks is favourable, although the anticipated gains are case-specific.

The present LCA study on standard and tailings-containing bricks followed the cradle-to-gate system boundary, which did not assess the impacts of bricks outside the defined systems, such as the potential releases of pollutants during use and end-of-life phases and eventually the recycling of used bricks. While the choice of system boundaries is valid according to NBN EN 15804:2012 + A2:2019/AC:2021 standard, following the environmental product declaration (EPD) guidelines, a further extension of system boundaries, including use and disposal stages, is necessary to avoid unintended environmental consequences beyond standard EPDs. For instance, the incorporation of waste materials containing leachable elements might transfer the contamination risk from the landfilling of traditional residues to the leaching of elements during materials use and further in the other downstream systems. Since tailings were generated from metal (Pb–Zn) production, some metal (loid)s are still present in the bricks and may escape in the subsequent life cycle stages, despite being constituted into bricks. On the other hand, the chosen allocation model does not account for credits of used bricks at the end of their life. Waste statistics from the Flemish Construction Confederation (VCB, 2021) show that the Flemish region has a high recycling share of CDW (at least 90%). This would imply that there is already a working collection and recycling system in place that can help minimize the pressure on natural resources (e.g., reprocessing demolished bricks to substitute other primary raw materials such as aggregates) and applies for both brick types. Additionally, the allocation model does not account for utilisation of tailings and thus reduction of the environmental risks of its emissions. Including these potentially avoided emissions would further decrease the impacts of tailings-containing bricks. Furthermore, no changes were modelled in this LCA study for the manufacturing stage. Further research might examine the effect of large-scale use of tailings on the manufacturing stage, such that trade-offs between lower energy requirements and potentially higher emissions to air involved in the firing process of tailings-derived bricks can be clearly identified.

4.3. *Recyclability of demolished mine waste-containing facing bricks*

In an end-of-life scenario, the demolished/crushed Plombières tailings-containing facing bricks showed a leachability of metal (loid)s below the limit values (Appendix H) in a preliminary assessment (batch leaching test) for potential landfill as inert waste (European Commission, 2003) and in a full assessment (column leaching test) for a second life scenario where shaped building ceramics can be demolished and recycled as aggregates for use in a new brick production or downcycled for cover layers, earth-rock filled dams, etc. (VLAREMA, 2012). It has been previously shown that the fired facing brick blends containing cleaned Neves Corvo waste rock materials (LM_5 and LM_7) did not comply with environmental regulations during service life (Appendix H) and cannot be commercialised. They also did not comply with a second life scenario due to leaching of As above regulatory limits, especially for the stored waste rock blend (LM_7) (Appendix H). In an hypothetical end-of-life scenario for the cleaned Neves Corvo waste rock bricks, they could only be disposed of in landfills for hazardous waste, once again due to leaching of As above limit values for landfilling as inert or non-hazardous waste (Appendix H).

Therefore, due to the low leachability of metal (loid)s, it can be concluded that the selected Plombières tailings is a ready-to-use alternative raw material for circular facing bricks, where apart from following the production processes and product quality criteria that are comparable with the standard, the environmental performance during service life, second life or end-of-life is not compromised.

5. Concluding remarks

The scope of this study was to evaluate the potential use of three (cleaned) sulphidic mining waste materials in a company-specific ceramic facing brick blend. This evaluation considered the production processes, product quality, and environmental performance of the fired test specimens during service life, 2nd life, and end-of-life scenarios, as well as a cradle-to-gate life cycle assessment of the best performing facing brick test blend.

As the Plombières tailings material is a fine-grained material with low sulphur and metal (loid) content, it does not require any mechanical and/or (bio)chemical pre-treatment before incorporation in a facing brick blend. The Plombières tailings-containing (20 and 40 wt%) test specimens showed very similar aesthetical and chemical properties and slightly better technical properties when compared to the standard. Moreover, the fired tailings-containing (40 wt%) test specimens were, like the standard test specimens, in compliance with the environmental performance tests for metal (loid)s leaching during service life (application as shaped building material), 2nd life (recycled or downcycled as aggregates), and at the end-of-life (landfilled as inert waste). The advantages of the use of Plombières tailings material in fired bricks were also confirmed by the environmental perspectives through the cradle-to-gate LCA.

On the contrary, both Neves Corvo waste rock materials (fresh and stored) required mechanical and chemical pre-treatment due to their high sulphur and metal (loid)s content. Comminution and froth flotation were performed on both waste rock materials in order to remove the high sulphur and metal (loid) content before incorporation in the facing brick blends. Nevertheless, facing brick test specimens using 20 and 40 wt% of cleaned Neves Corvo waste rock materials induced aesthetical problems, such as efflorescence and white-staining on the surface of the test specimens after drying and firing. On the test specimens with 40 wt% waste rock materials, chemical-environmental problems were found with excessive leaching of As during diffusion test (service life), meaning that such brick cannot be produced according to the Flemish environmental regulations.

This study demonstrated the added value of a complete characterisation of alternative raw materials, such as mining waste, in order to assess their potential in replacing primary raw materials in circular building products.

Uncited reference

citation(s) ‘Best, 2018’ has/have been changed to match the date in the reference list. Please check here and in subsequent occurrences, and correct if necessary.>, Salinas-Rodrí guez et al., 2017.

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EN 15935, 2012; ISO 10694:1995, 2012; ISO 14869-1:2001, ; ISO 17294-2:2016, ; NBN EN ISO 10304-1, 2009; NBN EN12457-2, 2002; .

CRediT authorship contribution statement

Francisco Veiga Simão: Formal analysis, Investigation, Data curation, Writing – original draft. **Hilde Chambart:** Supervision, Conceptualization, Validation, Writing – review & editing. **Laure Vandemeulebroeke:** Methodology, Resources, Validation. **Peter Nielsen:** Methodology, Resources, Validation, Writing – review & editing. **Lugas Raka Adrianto:** Investigation, Data curation, Software, Writing – original draft. **Stephan Pfister:** Methodology, Resources, Validation, Writing – review & editing. **Valérie Cappuyns:** Supervision, Visualization, Writing – review & editing.

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.

Data availability

Almost all data are given in the paper. The few data that are not given are confidential.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jclepro.2022.133118.

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