



# Solar Assisted Production of $\text{MgAl}_2\text{O}_4$ from Bayer Process Electrofilter Fines as Source of $\text{Al}_2\text{O}_3$

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## Abstract

Compared with conventional high-temperature methods based on electricity and fossil fuels, concentrated solar energy route offers distinct advantages in terms of mitigating emissions of contaminants and shortening processing times. Nevertheless, solar-based route also encounters challenges in producing significant quantities of materials, although the deployment of this technology is still conditioned by the limited investigation in the field. This study presents a novel high-temperature process based on solar energy to produce  $\text{MgAl}_2\text{O}_4$  spinel, which employs as source of  $\text{Al}_2\text{O}_3$  a waste from the aluminum industry: waste alumina fines from the Bayer process. First, mixtures were prepared by mechanical mixing in a molar ratio 1:1 in agreement with the  $\text{MgO}-\text{Al}_2\text{O}_3$  binary phase diagram. Then, synthesis of the  $\text{MgAl}_2\text{O}_4$  spinel was conducted by static experiments (5 min) with direct application of concentrated solar energy ( $1150 \text{ W/cm}^2$ ) at temperatures greatly exceeding  $1800 \text{ }^\circ\text{C}$  as reported by ANSYS software. Wastes from three Bayer process factories were studied, which exhibited after the synthesis process a good crystallinity. The carbon dioxide emissions avoidance would range from the 200 to 500 tons of  $\text{CO}_2$ /year in the case of a small plant producing 1000 tons/year to 5000 to 12,000 tons of  $\text{CO}_2$ /year in the case of a commercial plant producing 25,000 tons/year, thus contributing to mitigate climate change. The proposed process might lead to smaller volume of wastes in the aluminum industry, while the  $\text{MgAl}_2\text{O}_4$  may be used as raw material in the numerous fields based on the chemical, thermal, dielectric, mechanical and optical properties.

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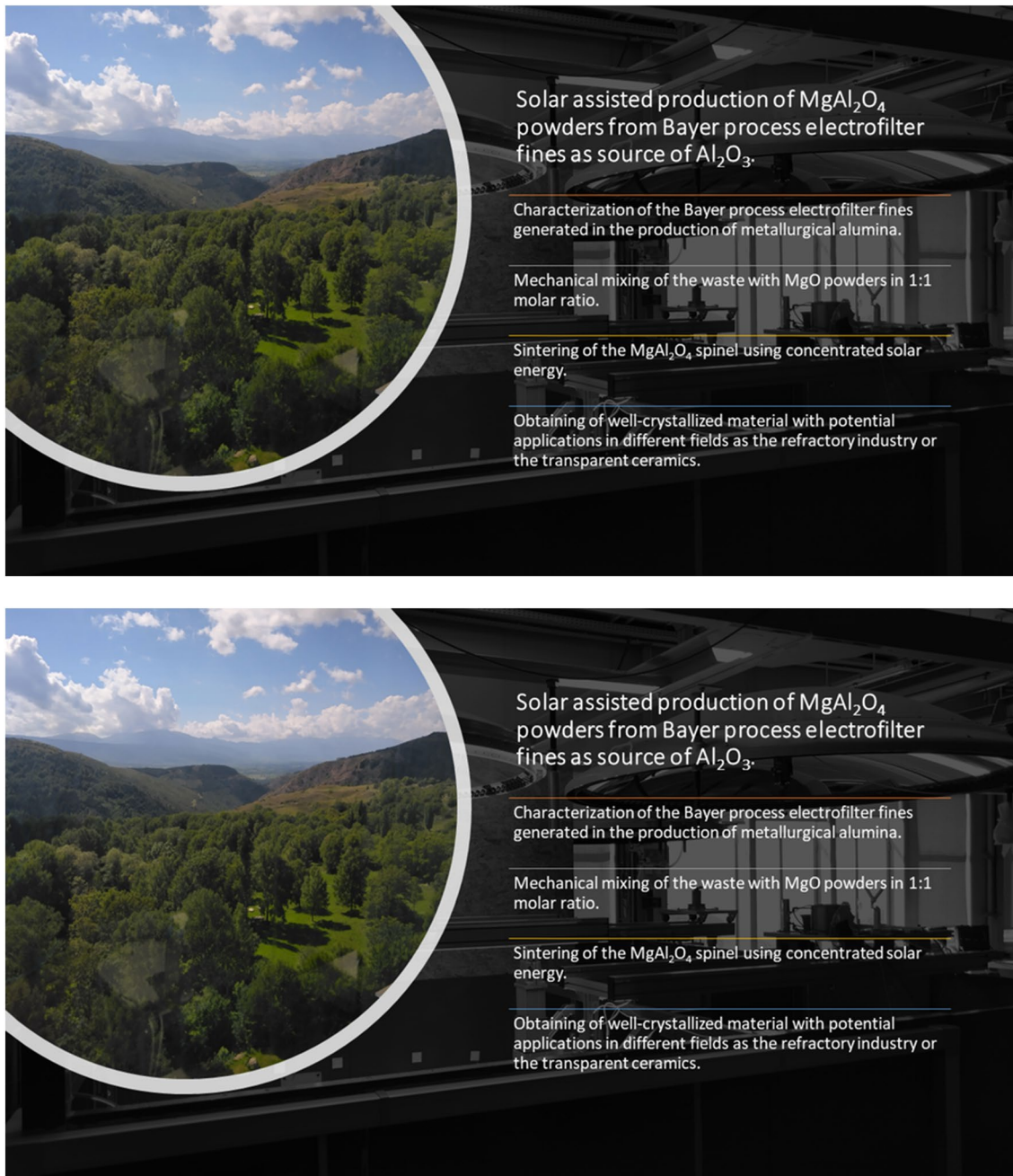
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## Graphical Abstract



**Keywords**  $\text{MgAl}_2\text{O}_4$  spinel · Concentrated solar energy · Metallurgy · Circular economy · Sustainability

## Introduction

Aluminum industry, accounting for a smelter production of 68 Mt in 2021 according to the *United States Geological*

*Survey*, is one of the most produced materials [1]. The production of aluminum is carried out by means of a process that involves two main stages, one, called Bayer process, which has as objective to produce alumina from bauxite

(consisting of mainly aluminum hydroxides with variable quantities of iron oxides, aluminum clays, kaolinite, and minor amounts of other oxides [2]) and the other, called Hall-Héroult process, which aims at producing aluminum by electrolysis in a molten electrolyte of cryolite. Focusing on the Bayer process, it involves several stages that include the pressure leaching of bauxite ore with aqueous soda solution, precipitation of aluminum hydrate (bayerite) from clean solution and calcination of the hydrate to produce alumina, which is later employed as raw material in the Hall-Héroult process.

Electrofilter fines are a residue generated in this process in quantities of around 5–8% of the final alumina product (approximately 80,000–100,000 tons for a 1.2 Mt alumina plants [3]). It consists of a mixture of different aluminas and alumina hydrates with small particle size that are collected as a residue in the electrofilter equipment. Different studies were conducted to find an application or extract valuable components from the electrofilter powders generated in the Bayer process, although there is still not any process with a massive implementation in the industry. This way, Bayer process electrofilter fines have been used in the production of ceramic materials as Tutic and collaborators, which obtained mullite ceramics from Bayer electrofilter fines and low kaolinite clay [4], and Sancho-Gorostiaga and others [5], which proposed the utilization of this waste in the manufacture of high temperature structural insulators. Other researchers tried to collect components from the residue as Okudan and collaborators, which attempted to recover gallium and aluminum [6]. For their part, Ayala and Pérez [7] investigated the removal of Se(VI) from aqueous solution using Bayer process electrofilter fines. Finally, other research in this line was conducted by Sancho et al. [8] and Ayala et al. [9], which recycled electrofilter fines as aluminum sulphate for water coagulant uses, Sancho and co-authors [3], which suggested that fines could be recycled as industrial quality aluminum sulphate, alums, and abrasives, and Sancho-Gorostiaga and colleagues [10], which investigated about the removal of copper from aqueous solutions using Bayer process electrofilter fines.

Solar energy, when it is adequately concentrated, offers enormous possibilities in metallurgy and materials science [11]. A recent state-of-the-art review of Fernández-González [12] makes a deep literature survey of the applications of solar energy in the field of materials. Our research group has made intensive work in the application of concentrated solar energy in the synthesis of materials: silicon-calcium [13], where the synthesis of this energy-intensive alloy used in deoxidizing of steels was attempted from mixtures of CaO and Si to avoid carbon dioxide emissions and energy costs, although it was produced in minor quantities associated to the atmosphere of the process; silicon-manganese [14], where the synthesis of this alloy typically used in the

production of steels was achieved from mixtures of manganese (IV) oxide and silicon; calcium aluminate cements [15], where the most relevant result was the synthesis of main components of the high alumina (> 75%) calcium aluminate cements using limestone as source of CaO for the process; operations in the field of iron and steels [16], where metallic iron was obtained from iron ore sinter fines reduced by coke with the heat support of solar energy; and, zirconia [17], which was obtained by solar thermal decomposition of zirconium silicate sand and subsequent separation of the  $ZrO_2$  by pressure leaching with NaOH of the silica. Focusing on the aluminum field, solar energy has been applied to reduce the environmental impact by using a zero- $CO_2$  energy. First investigations in this line can be attributed to J. P. Murray [18], who proposed the direct reduction of  $Al_2O_3$  to aluminum or aluminum-silicon alloy or reduction to AlN or  $Al_2S_3$  that could be more easily electrolyzed with non-consumable electrodes. Several studies were conducted from that moment in line with the production of aluminum by carbothermal reduction with more or less success in the obtaining of metallic aluminum [19–24]. Alternatively, Lytvynenko [25] proposed the utilization of solar energy in the obtaining of aluminum by electrolysis through the Hall-Héroult's process using the current of a solar battery and employing solar radiation to heat the cell. In the same line, other researchers proposed the calcination of the aluminum hydroxide to obtain alumina to feed the cell for the electrolysis of aluminum using solar energy [26, 27].

Nevertheless, apart from the potential application of solar energy in the synthesis of materials, there is a growing interest in its application to recycle or recover valuable components from industrial wastes, for instance in the extraction of copper and iron from copper slags [28] or iron from basic oxygen furnace slags [29], due to the possibility of heating without releasing pollutant. The research in this field focuses not only on the recovery of metals from slags but in the immobilization of contaminants, as filter dusts [30, 31] or soil and mine waste samples containing mercury [32–35]. Anyway, the application of solar energy in recycling or recovering metals is the most important line due to the lower temperatures required in the case of operating with them whether compared with ceramics. Therefore, other researchers aimed at recovering metals (Zn, Fe, Pb and Cu) from electric arc furnace dust (EAFD) and automobile shredder residue (ASR) [36, 37], extracting zinc from Waelz oxide produced during the recycling of galvanized steels [38] or used solar energy to melt aluminum scrap [39–42]. Nevertheless, as a consequence of the high temperatures that can be reached with the competition of solar energy (above 2500 °C [11, 12]) researchers have also attempted to recycle aluminum oxides. This way, studies guided by Padilla and collaborators [43] in the field of

aluminum waste were conducted to obtain alumina from boehmite prepared by sol–gel process from an aluminum waste that proceeded from the fine suction system used in the aluminum slag milling operation. The same authors, Padilla and researchers [44], attempted the production glasses also from aluminum wastes, which consisted in a powdered solid trapped in filter sleeves during the slag milling process of the aluminum tertiary industry. Apart from the aluminum residue, these researchers employed other wastes as eggshells and mussel shells as sources of calcium for the glass.

Magnesium aluminate compound is a spinel that is gaining interest in different applications due to the excellent chemical, thermal, dielectric, mechanical and optical properties [45]. Nevertheless, magnesium aluminate spinel is an excellent refractory oxide of great importance as structural ceramic due to the physical, chemical, and thermal properties at normal and elevated temperatures [46]. Different methods can be used to obtain the  $MgAl_2O_4$  spinel. These methods include sol–gel reaction [47], gelatin method [48], metal-chitosan complexation [49], thermal decomposition [50], hydrothermal [51], high-energy ball mill [52], microwave assisted combustion [53], organic precursor combustion [54], sonochemical [55], spray pyrolysis [56], spray drying [57], among other processes [47]. Anyway, the most habitual technique to synthesize this spinel is the solid-state reaction from powders of  $MgO$  and  $Al_2O_3$ , although the process has several disadvantages including long processing times, need for repetition of several calcination stages, requirement of high temperatures for sintering [46]. Some of these issues might be solved with the application of concentrated solar energy. For that reason, this manuscript proposes a novel method to synthesize  $MgAl_2O_4$  from Bayer process electrofilter fines at laboratory scale using concentrated solar energy as potential route to reduce carbon dioxide emissions.

## Materials and Methods

### Raw Materials

Three different Bayer Process Electrofilter Fines (BPEFs) were used as source of  $Al_2O_3$  in the obtaining of the  $MgAl_2O_4$  spinel, while the  $MgO$  was provided by an industrial supplier. The BPEFs were generated in Bayer process plants located in three different countries: Canada, Ireland, and Spain. The chemical composition was determined by inductively coupled plasma mass spectrometry, while carbon and sulphur contents were obtained by combustion with the equipment CS800 Eltra. Results for the BPEFs are collected in Table 1. The structure of the BPEFs was analyzed by X-ray diffraction, which reported different types of alumina (mainly  $\alpha$ - and  $\gamma$ -alumina, whose content was determined by means of the area below the X-ray diffraction pattern) with certain amounts of gibbsite ( $Al(OH)_3$ ). The carbon content gives a characteristic grey color to the sample. Mean particle size ( $d_{50}$ ) was determined by photo-sedimentometer Lumosed equipment. The water content in the samples is represented by the Moisture on Ignition (MOI, mass losses when heating the sample up to 550 °C for 2 h) and Lost on Ignition (LOI, mass losses when heating the sample up to 900 °C for 2 h). Additionally, the content in aluminum trihydrate (gibbsite) was determined by thermal analysis technique (Mettler-Toledo DSC822e), under a nitrogen atmosphere (50 ml/min) and at a heating rate of 5 °C/min to determine the endothermic peak corresponding to the hydrate water loss, where patterns with variable contents in gibbsite (from 0.5 to 50%) were employed to obtain the calibration line. This parameter is important because gibbsite is also a source of  $Al_2O_3$  for the spinel although before the reaction with the  $MgO$ , the aluminum trihydrate decomposes into  $Al_2O_3$  and  $H_2O$ , which leads to problems of decrepitation. Numerical values of the characteristics of the BPEFs are collected in Table 2.

**Table 1** Composition of the BPEFs of the three Bayer process factories

(%)	$Al_2O_3$	$Na_2O$	$SiO_2$	$Fe_2O_3$	CaO	$K_2O$	$TiO_2$	MgO	$Ga_2O_3$	C	S
Canada	87	0.70	<0.04	<0.04	0.03	<0.04	<0.01	<0.01	0.008	0.08	0.02
Ireland	92	0.46	<0.04	<0.04	0.02	<0.04	<0.01	<0.01	0.012	0.14	0.02
Spain	91	0.67	<0.04	<0.04	0.01	<0.04	<0.01	<0.01	0.012	0.14	0.02

**Table 2** Characteristics of the BPEFs of the three Bayer process factories

	MOI (%)	LOI (%)	$\alpha$ -Alumina	$\gamma$ -Alumina	Gibbsite (%)	$d_{50}$ ( $\mu m$ )
Canada	10.0	2.30	19.0	49.0	29.0	7.8
Ireland	5.3	1.60	17.0	65.5	14.5	5.3
Spain	6.6	0.7	33.0	32.2	18.0	8.7

Magnesia was provided by Grupo Peñoles (Mexico). The chemical composition of the magnesia was determined by X-ray fluorescence spectrometry. Results are collected in Table 3.

## Methods

Experiments were carried out in a vertical axis solar furnace located in Font Roméu-Odeillo-Via (France), which belongs to the PROMES-CNRS (PROcédés Matériaux et Energie Solaire-Centre National de la Recherche Scientifique). It consisted in a 1.5 m in diameter parabolic concentrator that is illuminated with flat heliostat, which has a sun tracking system. This way, a maximum concentration ratio of 15,000 in a focal point of  $\leq 1.5$  cm is achieved. This means that the maximum concentrated power would be 900 W for a  $1000 \text{ W/m}^2$  DNI (Direct Normal Irradiation), achieved with clean skies and parabolic concentrator. Experiments were conducted under a glass hood to avoid the deterioration of the parabolic concentrator with the powders that could leave the sample. Besides, air suction was applied to avoid the deposition of the powders in the glass hood, which would have been detrimental to the process. A scheme of the process appears represented in the Fig. 1.

The route chosen to manufacture the  $\text{MgAl}_2\text{O}_4$  spinel was the solid-state reaction. Mixtures were prepared in a 1:1 molar ratio according to the following chemical reaction:



which according to the binary phase diagram  $\text{MgO-Al}_2\text{O}_3$  is in the stoichiometric spinel zone. Sintering zone, according to Sarkar and Banerjee [58], occurs in the temperature range 1550–1650 °C, although the formation of the spinel occurs also at greater temperatures as it appears in the phase diagram.

The temperature of the spot generated by the concentrated solar energy was determined by finite element method-based software ANSYS. The dimensions of the crucible were 19.55 mm in inner diameter, thickness of 3.28 mm and height of 25.4 mm, manufactured in alumina. The crucible was filled with a mixture of powders of  $\text{Al}_2\text{O}_3$  and MgO in the proportion 1:1 molar. The following properties were considered for the thermal simulation: density, thermal conductivity, and specific heat of the different materials in agreement with the proportions. It is possible to see in Fig. 2a the scheme of the process with the dimensions used in the calculations. The analysis was carried out in a transient thermal module using the ANSYS Workbench application. To ensure the reliability of

simulation results, a finite element mesh convergence analysis was conducted. This entailed progressively refining the finite element mesh from a coarse (Fig. 2b (1) coarse mesh) to a finer resolution (Fig. 2b (2) refine mesh), resulting in an increased number of elements/nodes. Throughout this iterative process, temperature variations were monitored until reaching a point where the variations became negligible. Additionally, a finer mesh refinement was implemented specifically in the central region of the specimen, where the energy beam impacts. On another note, Fig. 2c and d show the simulations conducted with the software ANSYS for different spot size and, therefore, energy density. The dwell time was in both situations 5 min, with 5 min of cooling. Figure 2c shows the simulation done with a beam of 10 mm in diameter, which involves  $1150 \text{ W/cm}^2$  of energy density. This situation reported a maximum temperature of 1944 °C. On the other hand, Fig. 2d represents the simulation done with a beam of 15 mm in diameter, which involves  $550 \text{ W/cm}^2$  of energy density. This scenario gave a maximum temperature of 1676 °C.

Mathematical simulations carried out are useful to know the approximate temperature of the spot, but they are not adequate to understand the complete process. This way, mathematical simulations report that the temperature in the spot is sufficiently high to synthesize the  $\text{MgAl}_2\text{O}_4$  spinel. The simulations did not consider the consumption of the material as the process advances, it is necessary to consider that the final volume is 1/3 of the initial (the reasons are: powders manually compacted and sinter during the process, decrepitation, water losses, and flying of fines due to the air currents inside of the glass chamber). It is necessary to introduce in this point the thermocouple control. The thermocouple can be used to control the advance of the process as when the temperature starts to fall in the point where it is located (Fig. 1b), it is possible to assume that radiation is not producing any effect on the sample. Figure 3 serves as an example of the thermocouple measurements. The utilization of thermocouples inside of the sample is not a suitable strategy because they move and melt during the process.

There is not a control of the radiation applied to the sample (in other solar furnaces it is possible to employ a shutter to control the percentage of incident radiation that is applied to the sample). Within this line incident radiation during the experiments presented in this manuscript exceeded  $950 \text{ W/m}^2$ , which indicates that a power of around 0.9 kW was applied on a surface  $< 1.5$  cm in diameter.

The duration of the treatment was considered a crucial factor in the manufacture of the  $\text{MgAl}_2\text{O}_4$  spinel. Within this context, each experiment lasted 5 min. If

**Table 3** Characteristics of the MgO

MgO (%)	CaO (%)	SiO <sub>2</sub> (%)	Al <sub>2</sub> O <sub>3</sub> (%)	Fe <sub>2</sub> O <sub>3</sub> (%)	B <sub>2</sub> O <sub>3</sub> (%)	d <sub>50</sub> (μm)
98.5	1	0.2	0.15	0.13	0.01	< 45

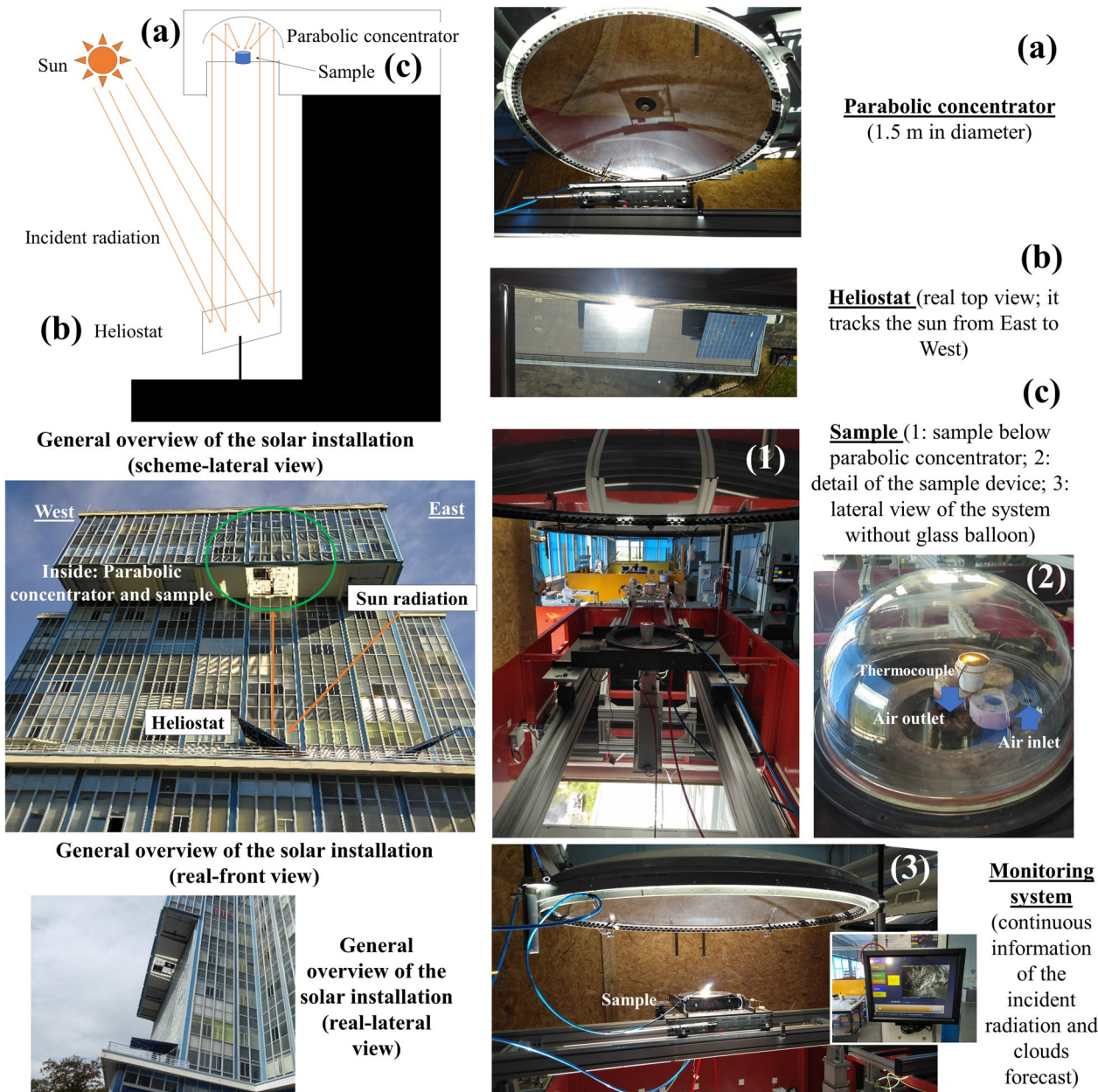


Fig. 1 Scheme and real images of the installation and devices used in the experimental work

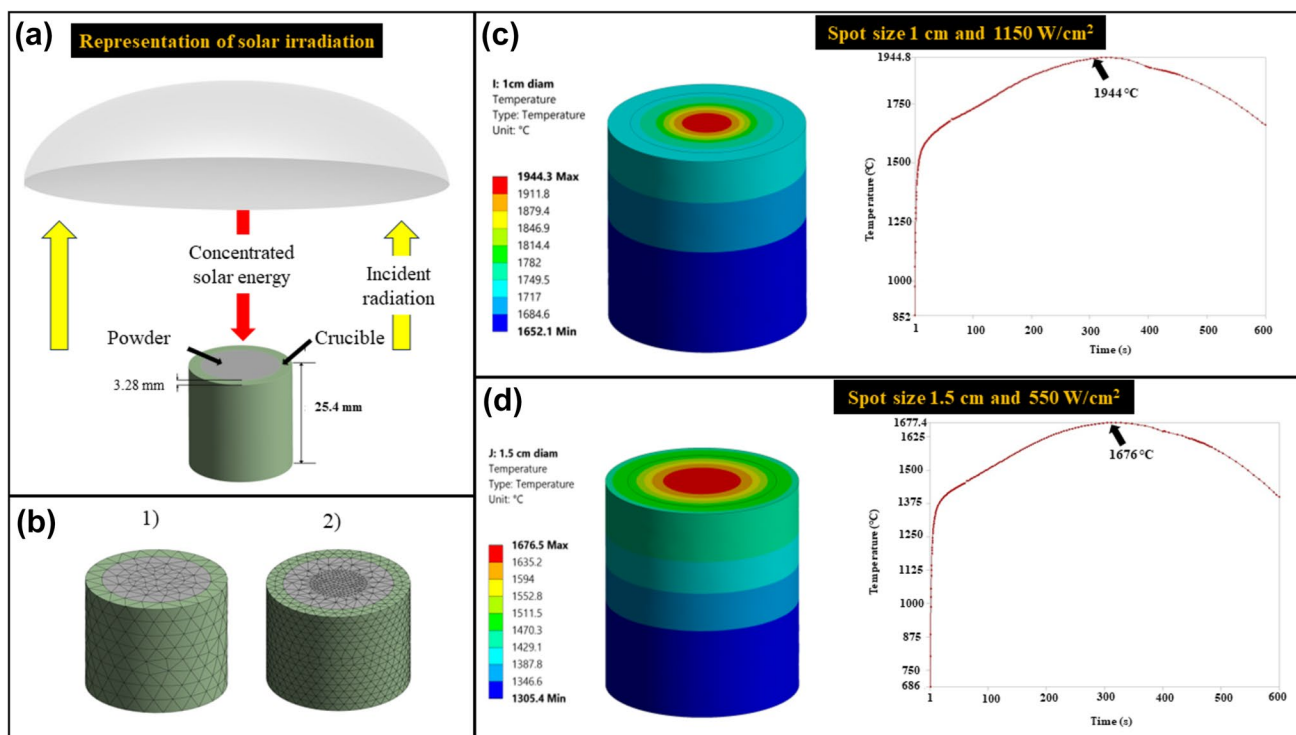
the operations of charging the crucible and cooling are included, the process would last approximately 20 min in total. It is necessary to consider that the process presented in this manuscript is based on the direct application of the solar radiation to the sample. Nevertheless, there are technologies to directly process powders in solar furnaces [12]. This might be following step that should be studied in the scalation of this technology up to industrial scale, as the direct processing of powders would significantly increase the productivity of the process.

## Results and Discussion

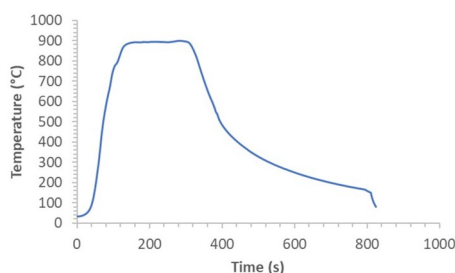
### Mass Differences

Table 4 collects the weight losses produced during the experiments, which are consequence of several reasons discussed in the following lines.

*Water:* this is the main reason of the mass differences resulted from the treatment of the samples with concentrated solar energy. Aluminum industry residue (BPEF)

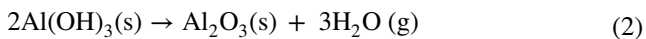


**Fig. 2** a representation of solar irradiation, b meshing of the crucible and charge c simulation carried out with a spot size of 1 cm and 1150 W/cm<sup>2</sup> and d simulation carried out with a spot size of 1.5 cm and 550 W/cm<sup>2</sup>



**Fig. 3** Thermocouple measurement, located in the point indicated in the Fig. 1B

was not subjected to any previous treatment, and it contains certain quantity of physical water resulted from the storage outdoors. This involves that moisture depending on the  $\gamma$ -alumina content was: 0.9% in Canada BPEFs, 2.6% in Ireland BPEFs and 0.4% in Spain BPEFs. Additionally, there is certain quantity of chemical water since the residue contains aluminum hydroxide that decomposes at temperatures of around 550 °C according to the reaction 2.



It is necessary to consider MOI measurements for Canada BPEFs were as high as 10%, while those corresponding to

**Table 4** Weight losses produced during the treatment with concentrated solar energy

	Initial weight (g)	Final weight (g)	Mass losses (%)
Canada 1	4.8	2.8	41.7
Canada 2	3.3	2	39.4
Canada 3	3	2	33.0
Ireland 1	3.2	2.4	25.0
Ireland 2	2.8	2.2	21.4
Ireland 3	2.9	2.3	20.7
Spain 1	3	2	33.0
Spain 2	3.8	2.7	29.0
Spain 3	3.4	2.8	17.6

the BPEFs of Ireland and Spain were 5.3% and 6.6%, in agreement with the greater or lower gibbsite content. Average mass losses were 38% in the case of Canada BPEFs, 22.4% in the case of Ireland BPEFs and 26.5% in the case of Spanish BPEFs. Therefore, losses are directly related to the gibbsite content in the residue, which is reasonable considering the water content in the hydroxide.

*Volatile matter:* the residue contains alkali metals, mainly Na<sub>2</sub>O, which according to the literature can evaporate at temperatures in the range 1500–1560 °C [59]. These authors

indicate that the presence of  $\text{Al}_2\text{O}_3$  promote the evaporation ratio ( $\text{MgO}$  also plays the same role, but it only slightly enhances the evaporation ratio). Carbon, which is in minor contents but gives the characteristic grey color to the mixture, is also susceptible of being burned at the temperatures of the treatment.

*Flying powders and decrepitation:* The presence of an air current inside of the glass balloon leads to powder flying at the beginning of the treatment of the sample with concentrated solar energy. This problem is also observed in the case of operating without glass hood. Nevertheless, one of the reasons of the mass losses is the decrepitation produced due to the fast-heating rate and the presence of water and hydrated compounds in the sample. It is necessary to consider that powders are very fine,  $d_{50} < 45 \mu\text{m}$ , which suggests that green compacts could be a suitable option to process these materials or employ a reactor like those proposed by other authors in the case limestone calcination [60] or  $\text{ZnO}$ - $\text{Zn}$  loop [61].

Quantitatively, mass losses associated with water (moisture + LOI + MOI, included the dehydration of the gibbsite) account for 28.50%, with chemical reactions (volatile

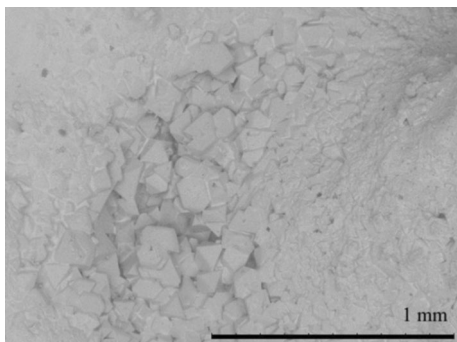
compounds + carbon combustion) are  $< 2\%$  and with projections, decrepitation and flying powders are 69.5%. The presence of impurities might play the role of fluxes, for instance sodium oxide is a common flux, but considering the excesses of energy employed in the proposed process the influence of the impurities in the reduction of the temperature is not relevant.

### Microstructural Characteristics

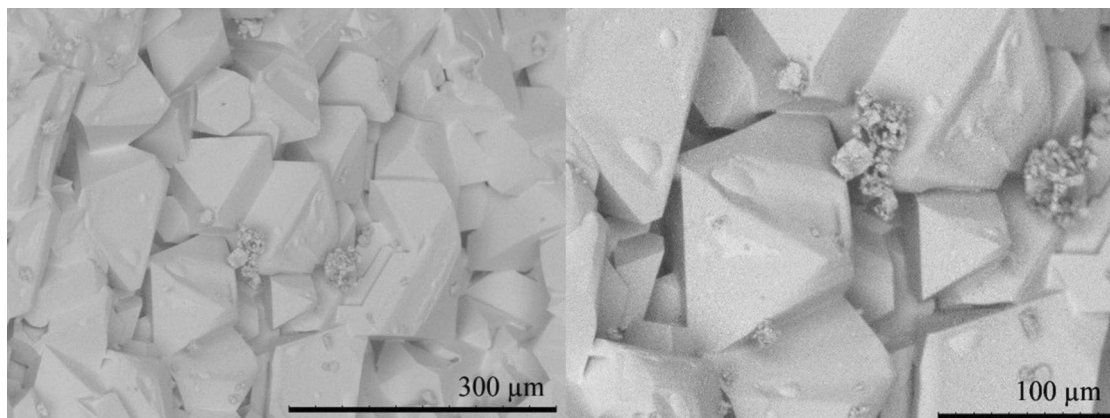
The product at the end of the treatment consisted in bulk material as it is showed in Fig. 4. This sample may be grounded and milled to the desired grain size for further applications. Likewise, other alternative may be the obtaining of sintered discs of  $\text{MgAl}_2\text{O}_4$  spinel, or the direct processing of powders as it was already anticipated. Despite the final product is obtained as a bulk material, it is crystalline as it is later reported in the X-ray diffraction analyses. In fact, if the Fig. 4 is observed, it is possible to check that crystals of  $\text{MgAl}_2\text{O}_4$  spinel are obtained at macroscopic scale. Figure 5a and b show images of the  $\text{MgAl}_2\text{O}_4$  spinel crystals at greater magnification.

Specimens were characterized by X-ray diffraction technique. This technique illustrates that the product obtained after the treatment is really  $\text{MgAl}_2\text{O}_4$  spinel with the suitable crystallinity. X-ray diffraction patterns are collected in Fig. 6 for the three  $\text{MgAl}_2\text{O}_4$  spinel prepared with the three BPEFs: Fig. 6a,  $\text{MgAl}_2\text{O}_4$  spinel with Canadian BPEFs; Fig. 6b,  $\text{MgAl}_2\text{O}_4$  spinel with Irish BPEFs; and Fig. 6c,  $\text{MgAl}_2\text{O}_4$  spinel with Spanish BPEFs.

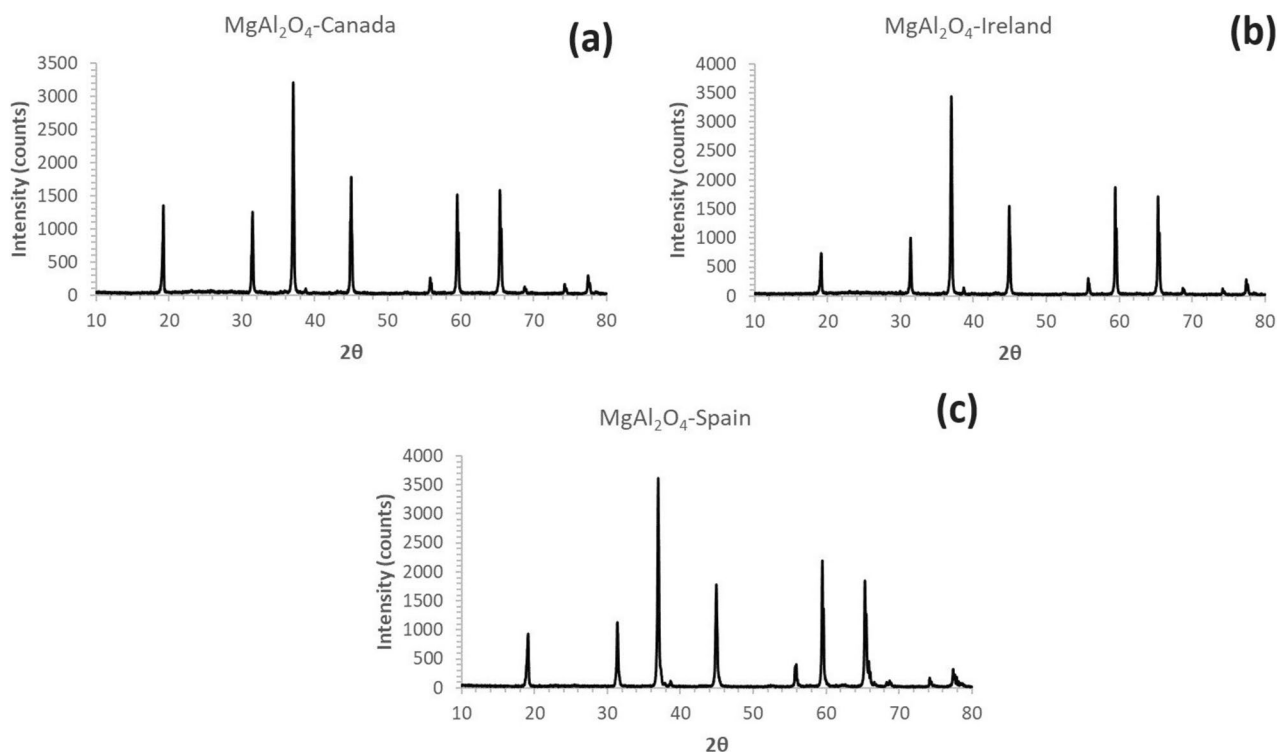
X-ray diffraction patterns report well crystallized specimens with no traces of the starting raw materials, which indicates that the treatment with concentrated solar energy allowed to obtain the  $\text{MgAl}_2\text{O}_4$  spinel, as it was expected from the phase diagram. Amorphous content in samples is: Canadian  $5.4\% \pm 2.0$ , Irish  $5.0\% \pm 1.9$ , and Spanish  $5.6\% \pm 2.0$ , determined with the X Powder Software.



**Fig. 4** Bulk  $\text{MgAl}_2\text{O}_4$  spinel sample obtained after the treatment with concentrated solar energy



**Fig. 5** Details of  $\text{MgAl}_2\text{O}_4$  spinel crystals



**Fig. 6** X-ray diffraction of MgAl<sub>2</sub>O<sub>4</sub> spinel using **a** Canadian BPEFs, **b** Irish BPEFs, and **c** Spanish BPEFs

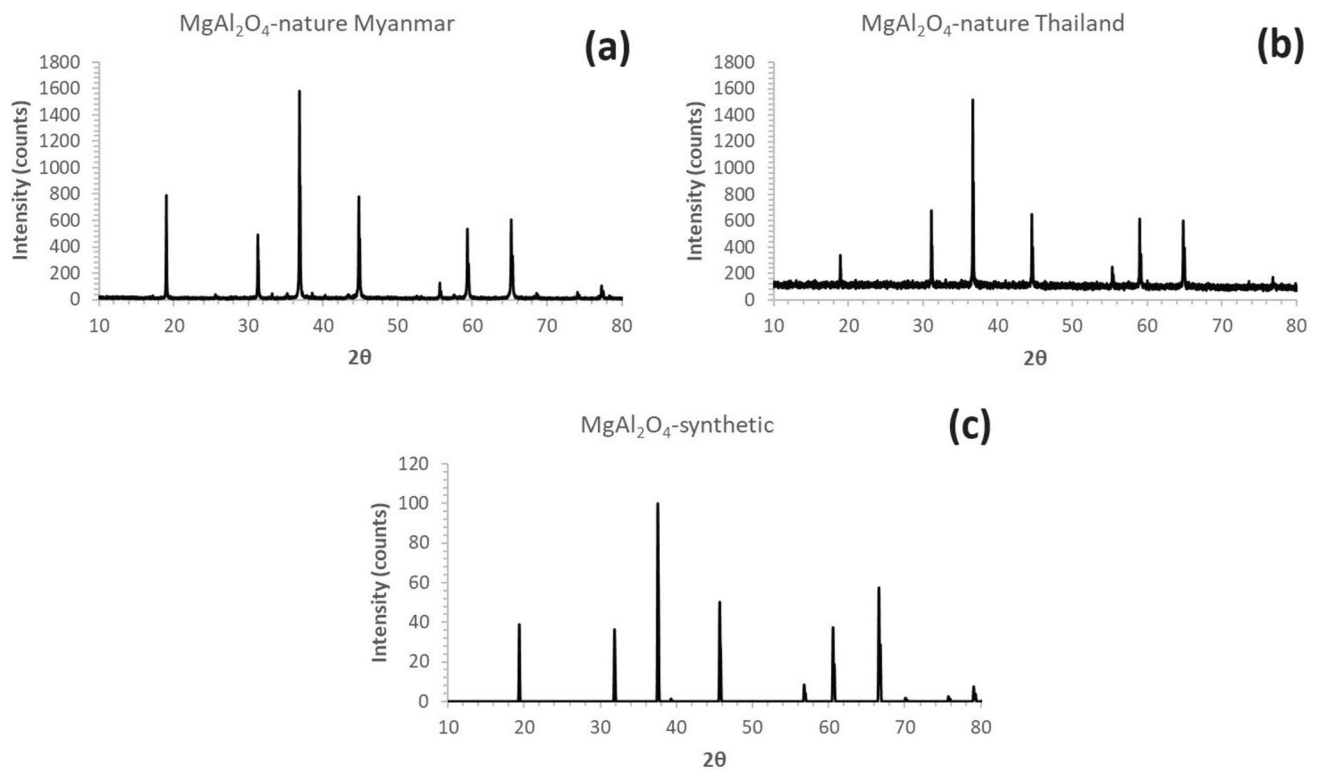
Figure 7 collects the X-ray diffraction patterns of various MgAl<sub>2</sub>O<sub>4</sub> spinels, either found in the nature as minerals or synthesized in laboratories. The form of the diffractograms analogous in the form to that obtained with the competition of concentrated solar energy.

The X-ray diffraction parameters  $2\theta$  and  $d$ -spacing for the  $hkl$  planes are compared with those of the reference work of Paterson and researchers [62], and some other MgAl<sub>2</sub>O<sub>4</sub> spinels found in the nature and synthesized in laboratory. This allows checking the quality of the obtained MgAl<sub>2</sub>O<sub>4</sub> spinel. Results are collected in Table 5 for the positions of the peaks and Table 6 for the  $d$ -spacing of the  $hkl$  planes. There is significant approximation of the values of the solar synthesized MgAl<sub>2</sub>O<sub>4</sub> spinel to those obtained by Paterson and co-authors [58], as well as to those found in the nature and, particularly to that synthesized in the laboratory, which is evidence of the well crystallization of the spinel obtained by means of concentrated solar energy using Bayer process electrofilter fines as source of alumina.

### Energy Efficiency and Technical Feasibility

Solar energy is a powerful source of heat that can be used in the obtaining of energy intensive products without carbon dioxide release in short times derived from the fast heating and cooling rates, as it is possible to see in Figs. 2 and 3. The synthesis of MgAl<sub>2</sub>O<sub>4</sub> spinel is an energy intensive process,

which is usually carried out at temperatures within 1550 and 1650 °C, although it is possible to obtain it at lower temperatures (> 550 °C) with prolonged permanence at these temperatures (> 4 h). Ping et al. remarked that the time for the synthesis of the MgAl<sub>2</sub>O<sub>4</sub> spinel is a relevant issue [46]. Within this line, routes proposed at laboratory scale by different authors are still time consuming and, therefore, synthesizing the spinel in short times is still a challenge. The low temperature synthesis by sol–gel process proposed by Pei and researchers [64] lasts at least 6 h, the process of modified sol–gel process proposed by Sanjabi and Obeydavi [65] also lasted at least 6 h, Meng and colleagues [66] indicate that their process took about 3 h. On the other hand, Bocanegra and co-authors [67] proposed different routes of synthesis that took > 36 h (ceramic method), > 48 h (mechanomechanical synthesis) and > 30 h (co-precipitation). In the field of mechanomechanical synthesis too, Kong et al. [50] required more than 14 h to sinter MgAl<sub>2</sub>O<sub>4</sub> spinel, with 12 h of milling in planetary ball milling and 2 h of sintering in furnace. Zhang in the hydrothermal route, required more than 31 h to synthesize the spinel [49]. On the other hand, Bai and researchers [68] employed more than 4 h in the combustion method. Citric acid–ethylene glycol route was used for the synthesis of MgAl<sub>2</sub>O<sub>4</sub> nano-powder, involving a synthesis time of 40 h [69]. The solar process could be competitive with microwave sintering, as irradiation lasts less than 1 h [51], although the process has the inconvenient



**Fig. 7** X-ray diffraction of MgAl<sub>2</sub>O<sub>4</sub> spinel found in nature (Myanmar) (a), nature (Thailand) (b), and synthesized in laboratory (c)

**Table 5** Position of the peaks in the X-ray diffraction pattern of solar MgAl<sub>2</sub>O<sub>4</sub> spinel compared with those obtained by Paterson et al., (1991) [58] and some other spinels either found in the nature or synthesized in the laboratory

	MgAl <sub>2</sub> O <sub>4</sub> -from Pein Pyit, Mogok, Myanmar (natural) [58]	MgAl <sub>2</sub> O <sub>4</sub> -from Bo Phloi, Kanchoutabori Province, Thailand (natural) [63]	MgAl <sub>2</sub> O <sub>4</sub> — (synthetic) [58]	MgAl <sub>2</sub> O <sub>4</sub> -Canadian BPEFs	MgAl <sub>2</sub> O <sub>4</sub> -Irish BPEFs	MgAl <sub>2</sub> O <sub>4</sub> -Spanish BPEFs
2θ	19.02		19.37	19.183	19.103	19.083
	31.28	31.11	31.89	31.446	31.386	31.366
	36.85	36.65	37.58	37.048	36.968	36.968
	38.55		39.32		38.688	
	44.81	44.56	45.72	44.990	44.930	44.950
	55.64	55.33	56.82	55.973	55.793	55.833
	59.34	59.01	60.61	59.574	59.494	59.514
	65.21	64.84	66.65	65.436	65.376	65.376
	68.60		70.14	68.917	68.777	
	74.09	73.65	75.79	74.298	74.238	74.238
	77.30	77.90	79.11	77.519	77.459	77.459

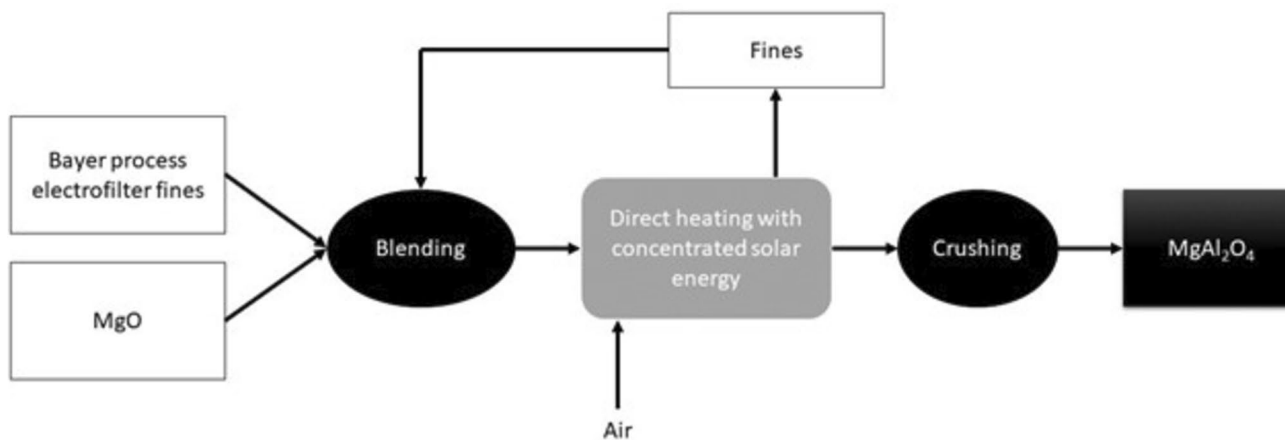
of using electricity for the microwave equipment. From the perspective of synthesis, Tripathy and Bhattacharya [70] indicated that it is still an important challenge to synthesize mesoporous MgAl<sub>2</sub>O<sub>4</sub> in a single step under rapid and economically viable approach. Their process required only 2 h to be completed by the flash pyrolysis route. Nevertheless, the process proposed in this manuscript might be seen as a competing alternative to produce MgAl<sub>2</sub>O<sub>4</sub> spinel in a single

step. The flow diagram of the solar process is presented in the Fig. 8.

It is important to remark that process proposed in this manuscript at laboratory scale required a maximum of 15 min for the process in the solar furnace. Even when the blending and crushing might be time consuming, the proposed route might represent a competitive alternative to produce the MgAl<sub>2</sub>O<sub>4</sub> spinel.

**Table 6** D-spacing of the hkl planes of the peaks in the X-ray diffraction pattern of solar  $\text{MgAl}_2\text{O}_4$  spinel compared with those obtained by Paterson et al., (1991) [58] and some other spinels either found in the nature or synthesized in the laboratory

	hkl	$\text{MgAl}_2\text{O}_4$ -from Pein Pyit, Mogok, Myanmar (natural) [58]	$\text{MgAl}_2\text{O}_4$ -from Bo Phloi, Kan-choutabori Province, Thailand (natural) [63]	$\text{MgAl}_2\text{O}_4$ — (synthetic) [58]	$\text{MgAl}_2\text{O}_4$ -Canadian BPEFs	$\text{MgAl}_2\text{O}_4$ -Irish BPEFs	$\text{MgAl}_2\text{O}_4$ -Spanish BPEFs
d-spacing	111	4.6706		4.5830	4.6231	4.6423	4.6471
	220	2.8602	2.8743	2.8065	2.8426	2.8479	2.8496
	311	2.4392	2.4512	2.3934	2.4246	2.4297	2.4297
	222	2.3353		2.2915		2.3255	
	400	2.0224	2.0324	1.9845	2.0133	2.0159	2.0150
	422	1.6513	1.6595	1.6203	1.6442	1.6464	1.6453
	333	1.5569	1.5646	1.5277			
	511	1.5569	1.5646	1.5277	1.5506	1.5525	1.5520
	440	1.4301	1.4371	1.4033	1.4252	1.4263	1.4263
	531	1.3674		1.3418	1.3631	1.3638	
	620	1.2791	1.2854	1.2551	1.2756	1.2764	1.2764
	533	1.2337	1.2398	1.2105	1.2304	1.2312	1.2312

**Fig. 8** Flow diagram of the solar process to produce  $\text{MgAl}_2\text{O}_4$  spinel

The processing times might be significantly reduced as well as the productivity of the process might be increased in further steps aimed at the scalation of the process. The direct application of the solar energy, as a process like that conducted in electric furnaces where the heating is punctual, is a potential alternative to produce  $\text{MgAl}_2\text{O}_4$  spinel although research conducted with other materials-processes might be potentially applied in this case. The production of lime by calcination of limestone and the thermal decomposition and reduction of zinc oxide with solar energy have been significantly studied using solar for the process [12]. The first investigations in this line were conducted by direct application of the solar beam to the samples in a laboratory scale but soon research acquired a greater dimension and investigators started to think about the increase of the productivity. Within this line, reactors are a key point and, therefore, have been significantly reviewed in the literature [71–73].

However, the importance of directly processing powders might be considered as one of the key points. Pilot plant scale studies have already validated the possibility of processing powders of limestone to produce lime, or reducing different metal oxides to obtain metal fuels, where the Paul Scherrer Institute has played an important role in the last 30 years [74]. Nevertheless, these indirectly heated reactors have never been applied in the synthesis  $\text{MgAl}_2\text{O}_4$  spinel. Thus, the following investigations in this line should be oriented to the synthesis of the compound in the form of powders, which would reduce even more the processing times as the steps of blending and crushing may be eliminated. There is not too much information about the world production-consumption of  $\text{MgAl}_2\text{O}_4$  spinel. Ganesh and collaborators indicated that the world consumption of spinel exceeded the 205,000 tons in 2004 [75]. This production, updated to current levels of consumption, might be easily covered with 10

solar factories considering that the conditions of production proposed by Meier and coauthors to produce lime (plant design of 25 MW<sub>th</sub> operating for around 2700 h per year to produce 27,000 tons) [76] are applicable in the case of the MgAl<sub>2</sub>O<sub>4</sub> spinel synthesis, although other researchers, as Purohit and researchers [77], increase the capacity of production (in this case for iron nuggets) to 200,000 tons/year. These technoeconomic studies, together with others reported in the review of Fernández-González [12] for different processes, suggest that the deployment of this technology at larger scale is only related with economic issues due to the lack of a plant operating for long periods of time under real conditions. Once validated in a real environment that it is possible to reach such capacities of production, plants to produce different materials will appear in countries with the suitable sun conditions.

Regarding the environmental issues, solar energy has a great potential in reducing carbon dioxide emissions. The energy demand required in the synthesis of the MgAl<sub>2</sub>O<sub>4</sub> spinel was determined by means of the software HSC Chemistry 5.1. Calculations include heating from the room temperature (25 °C) to the synthesis temperature (assumed to be 1700 °C) and energy required for the formation of the compound. It is important to indicate that the formation of the spinel occurs partially through intermediate decomposition of the gibbsite with a Al<sub>2</sub>O<sub>3</sub> to MgO molar ratio 1:1. Therefore, it is important to consider the gibbsite content in the thermodynamic calculations. Results are collected in the Table 7 for each residue of the aluminum industry, which indicates the energy required from the theoretical point of view.

Concentrated solar energy was directly applied to the sample. Power during the experiments was 900 W for a Direct Normalized Irradiation of around 1000 W/m<sup>2</sup> and a parabolic concentrator of 1.5 m in diameter with a concentration ratio of 15,000, a distance to the focal point of 65 cm, a maximum angle of incidence of 56° and a focal point of 1 cm in diameter. Therefore, the energy consumed during the experiments, which lasted for 5 min, is approximately 0.075 kWh to produce 2.4 g (31.25 kWh/kg, 112,500 kJ/kg). The

efficiency of the process ( $\eta$ ), defined as  $\eta = (E_{\text{Spinel}}/E_{\text{Total}}) \cdot 100$ , is < 5% in this case. In fact, Flamant and coauthors estimated that the energy conversion efficiency from the primary energy source to the useful energy beam reaches the 60% in the case solar furnaces [78]. It is necessary to remark at this point that there was not a strict control of the energy requirements in the process proposed in this manuscript as the objective was to demonstrate the feasibility of synthesizing the MgAl<sub>2</sub>O<sub>4</sub> spinel.

The potential reduction in carbon dioxide emissions was calculated. The efficiency of the electric arc furnace was assumed to be in the range 40–75% [79], which would be the energy source replaced by the solar energy. The plant sizes were assumed to be of 1000, 5000 and 25,000 tons MgAl<sub>2</sub>O<sub>4</sub> spinel per year, in agreement with the reference plant production sizes proposed by Meier and colleagues [76] for limestone calcination. Moreover, the carbon intensity of electricity in Spain in 2022 was 217 g CO<sub>2eq</sub>/kWh, according to the Energy Institute Statistical Review of World Energy. Results of the carbon dioxide avoidance are collected in Table 8. It is possible to see that the utilization of concentrated solar energy in the production of MgAl<sub>2</sub>O<sub>4</sub> spinel would promote a significant reduction in the greenhouse gas emissions and can contribute to the energy transition in the field of raw materials production for the manufacture of advanced ceramics.

## Conclusions

The synthesis of ceramic materials still involves processes that generate significant quantities of greenhouse gases, thus contributing to the climate change. Within this context, solar energy, when it is adequately concentrated, emerges as a suitable alternative for high temperature processes. Therefore, this renewable energy source has been applied in the synthesis of MgAl<sub>2</sub>O<sub>4</sub> spinel, where the source of alumina was a residue from the aluminum metallurgy called Bayer process electrofilter fines.

**Table 7** Thermodynamic calculations for the formation of the MgAl<sub>2</sub>O<sub>4</sub> spinel

Reaction	Energy demand (kJ/kg MgAl <sub>2</sub> O <sub>4</sub> )		
MgO + Al(OH) <sub>3</sub> → MgAl <sub>2</sub> O <sub>4</sub> + H <sub>2</sub> O	6047.7		
MgO + Al <sub>2</sub> O <sub>3</sub> → MgAl <sub>2</sub> O <sub>4</sub>	1979.3		
Energy demand (kJ/kg MgAl <sub>2</sub> O <sub>4</sub> )			
Canada	Ireland	Spain	
3159.1	2569.2	2711.6	

**Table 8** Carbon dioxide emissions avoidance considering the data collected in Table 7

	Efficiency (%)	Canada	Ireland	Spain
Energy demand (theoretical) (kJ/kg)		3159.1	2569.2	2711.6
Energy demand (real) (kJ/kg)	40	7897.8	6423.0	6779.0
	75	4212.1	3425.6	3615.5
Plant size (t/y)		Energy demand (kWh)		
1000	40	2,193,835.1	1,784,168.1	1,883,057.1
	75	1,170,028.7	951,556.3	1,004,306.4
5000	40	10,969,175.4	8,920,840.5	9,415,285.3
	75	5,850,143.6	4,757,781.6	5,021,531.8
25,000	40	54,845,877.2	44,604,202.4	47,076,426.6
	75	29,250,717.8	23,788,907.9	25,107,659.0
Plant size (t/y)		CO <sub>2</sub> emissions avoidance (t/y)		
1000	40	476.1	387.2	408.6
	75	253.9	206.5	217.9
5000	40	2380.3	1935.8	2043.1
	75	1269.5	1032.4	1089.7
25,000	40	11,901.6	9679.1	10,215.6
	75	6347.4	5162.2	5448.4

The synthesis of the spinel was conducted in very short times, not exceeding the 5 min under the action of the solar beam. The solar spot was at temperatures clearly exceeding 1700 °C, as it was reported by mathematical simulations, which is sufficient for the formation of the compound. The formation of the MgAl<sub>2</sub>O<sub>4</sub> spinel occurred partially through an intermediate process of decomposition of the gibbsite.

The fine particles of the residue together with the high heating rates and the water content produced certain mass losses that in a possible industrial process could be collected and sent back to the beginning of the treatment. Within this context, the direct application of the solar energy might be a technology that imitates the processes conducted in the electric arc furnaces in terms of direct application of the heat, although the processing of powders in indirectly solar heated furnaces might be a suitable alternative to directly process powder and reduce even more the processing times.

There is neither difference in terms of crystallinity nor phases when considering the three residues: Canada, Ireland, and Spain. The direct application of the solar radiation to the sample and the temperatures in excess to those required for the synthesis of the spinel hinder the possible influence of impurities in terms of reducing the synthesis temperature.

Three different scenarios were analyzed to evaluate the potential reduction in the carbon dioxide emissions. The values ranged from the 200 t/year in the case of small plants (1000 tons/year) to 12,000 t/year in the case of commercial plants (25,000 tons/year). This also translates into economics in terms of reducing the impact of the CO<sub>2</sub> emissions taxes.

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## Declarations

**Conflict of interest** The authors have no competing interests to declare that are relevant to the content of this article.

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