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Review article

Design of aluminosilicate refractory matrices in the context of climate change: sustainability approach

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ABSTRACT

The work proposes a cold-setting approach for designing and producing aluminosilicate refractories that save energy and emit low levels of CO_2. This process is based on shrinkage-free theory and the management of the expansion coefficient of refractory oxides from natural raw materials. Refractory composites can be designed to withstand high temperatures and thermal gradients. Refractory matrices based on Al-rich kalsilite, mullite, tialite and mullite-cordierite are designed with alternative cold-setting processes to achieve the desired porosity for dense and porous refractories. The formation and consolidation mechanisms of cold-setting refractory matrices are presented, demonstrating the role of colloidal silica and alumina in partially replacing an alkali-based colloidal solution. The optimum microstructure was accounted for the improvement of refractoriness, shrinkage and thermal resistance.

1. Introduction

Industrial development is defined by the science and technology of refractory materials, and this will remain true in the future. Aluminosilicate refractories are the primary choice for aluminium cell bottom linings, iron and steel production, and many other industrial applications, thanks to their worldwide availability, relatively low cost, and proven performance [1]. Unfortunately, the ceramic route used to produce aluminosilicate refractories significantly contributes to greenhouse emissions, air pollution and high energy consumption [2]. The aim of the cold refractory process is to ensure free shrinkage, moisture control, spalling and cracking resistance when the materials are in use. In order to reduce the 400 Mt of CO_2 emitted by the ceramic industries each year, and to protect the environment and ensure sustainable development for future generations, it is necessary to significantly reduce these emissions.

Nowadays, with advancements in technology and processing methods, it is reasonable to question whether aluminosilicate refractories require treatment at high temperatures (>1400-1500 °C) to produce high-performance products. The answer to this question refers to the possibility of achieving high-performance, free-shrinkage refractory composites using a cold-setting process [3]. The cold-setting process includes selecting and treating the raw materials, and moulding of the resulting mixtures. Selecting and treating the raw materials ensures refractory oxides with free shrinkage and good thermal stability, while the moulding process controls dimensional stability and the connection between the particles. These allowing for desired porosity, pore interconnectivity and microstructure. The cold-setting refractory process enables the shaping of smaller monolithic pieces with fewer defects, while ensuring volume stability and high strength at room and high temperatures [4,5].

The main raw materials used in the production of aluminosilicate refractories include kaolin, alumina hydrates, aluminosilicate hydrates and others such as MgO- and TiO_2-based materials. Dehydroxylation of these raw materials at temperatures of around 500–600 °C produces refractory oxides that can act as binders in cold-setting refractories thanks to particle optimisation and treatments. The refractory oxides would transform into refractory phases at high temperatures without

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shrinking, cracking or spalling, as the cold-setting skeleton is made up of refractory aggregates (such as kyanite and corundum) that are designed using particle grading to optimise the porous network and achieve desired microstructure. The cold-setting process would enable refractory industries to play a central role in the global transition to sustainable development, with CO_2-neutral production of steel, glass, cement, non-ferrous metals and chemicals. Refractory technology is essential for the entire industry.

The fundamental functions of refractories include: (i) providing a protective barrier to prevent thermal erosion and chemical corrosion by hot media such as flue gases, liquid metals, liquid slag and molten salts; and (ii) insulating the vessel [1]. Refractory materials are commonly used in furnaces, kilns, burner blocks and boiler works in various production lines (see Fig. 1) [6–8].

This review examines the strategies to reduce energy usage and toxic emissions through innovative processing techniques that explore shrinkage, particle grading and moulding methods to achieve zero shrinkage and an optimum microstructure capable of withstanding high temperatures. A descriptive analysis of novel sustainable alternatives indicates solutions for developing green refractories that can make significant contributions to achieving the Sustainable Development Goals as established by the United Nations. The focus is on technologies and strategies with significant potential to promote energy savings and reduce carbon emissions. The measures have been grouped into three main categories: (i) equipment level, (ii) plant level, and (iii) outer plant level. The implementation of innovative measures to improve energy efficiency reveals itself as a potential field of research, not only for reducing energy consumption, but also for achieving clean and low-carbon sustainability objectives at EU and global levels [4].

2. State of the art of the aluminosilicate refractories

2.1. Challenge of energy and toxic emissions

Ceramic materials are energy-intensive, accounting for a large proportion of the end product's cost. The fundamentals justifying the necessity to develop sustainable options for refractory materials are given here.

Given their contribution to global warming, refractories can today be considered a 'necessary evil'. Although refractories are among the most technologically advanced branches of the ceramics industry, emissions from their manufacture represent almost 20% of the 400 Mt CO_2 emitted each year. It is estimated that emissions linked to natural gas used for ceramic firing amount to 250 kg of CO_2 per tonne of ceramics. The annual global energy consumption for firing ceramics using natural gas is estimated at 182 TWh [2]. Emissions from refractories, or from ceramics in general, refer to the thermochemical transformations of the raw materials and fossil fuels used in manufacturing. In addition to CO_2, thermal processes in ceramics release sulphur, nitrogen oxides, fluorine and chlorine ([1]; [10]). If gas is used to perform temperatures up to 1850 °C, some refractories and others technical ceramics manufactures employ electric arcs for high firing temperatures that can reach up to 2700 °C. A significant proportion of the energy used in refractory manufacturing is allocated to drying, firing, and cooling processes, with the firing stage accounting for over 75% of the total energy cost [11]. The studies from Manrique et al. [12] evidenced that clay and ceramic floor tiles require 940 kWh per tonne, whereas ceramic products used in electrical applications, including high-tech refractories, require up to 5830 kWh per tonne. Concerns regarding the environmental impact of refractories and ceramics are also related to heat loss [13,14]. In fact, studies have shown that only 15-20% of the energy input is used for firing in kilns. The rest is lost through cooling stacks (30–35%), flue gas stacks (20–25%), kiln walls and vaults (10–15%), and fired bodies [13, 14]. In an intermittent kiln, firing occurs in batches and the fire is allowed to die out. The products are then cooled inside the kiln after the firing process. In a continuous kiln, the fire burns continuously and materials are heated, fired and cooled simultaneously in different parts of the kiln.

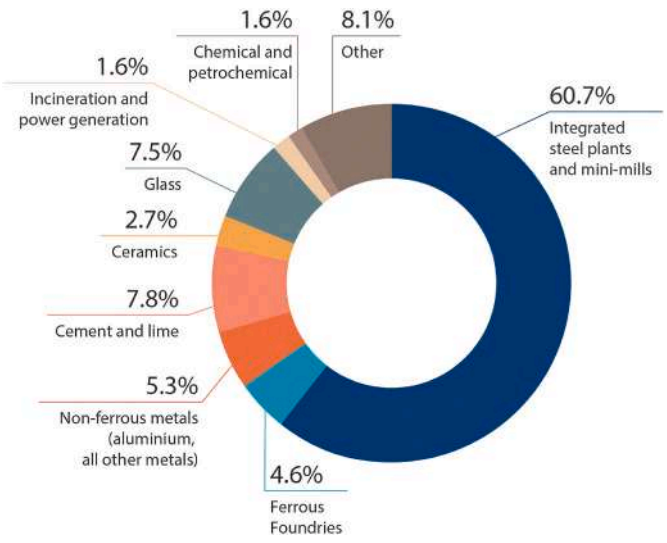


Fig. 1. The use of refractory materials in the different industries [9].

The emissions associated with the thermal processes of ceramic and refractory materials represent 90% of the total emissions from their manufacturing processes. These emissions are not limited to CO_2. The chemical composition of the raw materials, the gases used for firing, and the specific firing cycle affect the emissions. Apart from the carbonisation of organic matter, fluorine emissions are observed at 600 °C due to clay mineral dehydroxylation [2]. Above 900 °C, the fluorine emissions are linked to the decomposition of fluorite (CaF_2). Sulphur may also be emitted if pyrite is present in the raw materials alongside with organic matter [2].

2.2. Innovation and positive factors for sustainable processes

Since the primary role of refractory materials is to insulate, they do not have to be produced through firing. Nowadays, alternative manufacturing processes are key to maintaining the important role of refractory materials in supporting industries and technological development. Technological innovations and sustainable processes include identifying alternative raw materials, such as recycled materials, and processes that reduce the use of fossil fuels during high-temperature firing. Other innovations include emerging technologies, simulation and modelling, and prediction of correlations between formulations, properties and performance (Fig. 2). The driving force behind changes in refractory technologies has been to improve process technology while still ensuring thermal stability at high temperatures. It has also become necessary to protect the environment, and therefore people, from unhealthy waste and emissions. The initial ideas for producing sustainable refractories focused on alternatives to firing, such as using plastic clay as a binder to substitute ceramic bonds in refractories [1].

Knowledge of the intrinsic properties of raw materials enables the design of complex matrices with predictable characteristics. In the case of refractories, formulations can be designed according to the operating temperatures and the expected functionality of the refractory itself. Aluminosilicate raw materials, such as clays and bauxite, were the primary ingredient in 19th-century refractories, which were produced by hand [1]. Between 1900 and 1920, machine pressing was introduced alongside dry pressing in the UK and stiff mud in the USA [1]. Wire cutting was also used in the UK with clay as the raw material, to which 12-15% water was added (Lee and Moore, 1998). This made it possible to increase production from 5000 bricks per day to the same amount per hour, resulting in more regular, homogeneous and uniform bricks. However, basic and carbon bricks continue to be made through dry

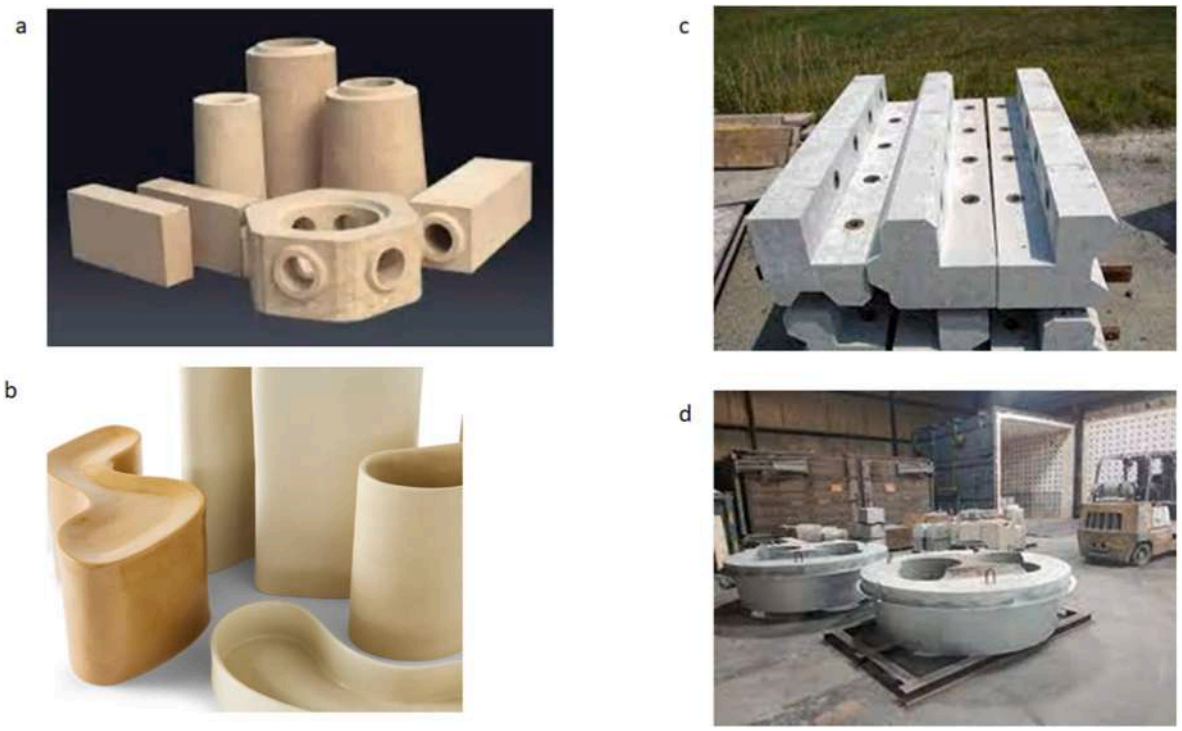


Fig. 2. Images of refractory materials from traditional fired bricks (a), castables (b) recent development of castables (c and d).

pressing [1]. Advances in science and technology have fully automated the production of refractory bricks with increased pressure and vacuum pressing. The development of carbon-bonded refractory bricks in the 1970s was the first step towards cold setting refractories [15]. Researchers and industries have taken inspiration from conventional concretes in order to design and develop refractory castables (Table 1). Today, the term 'refractory castables' refers to cold-setting refractories. Various setting agents, deflocculants and other additives are used to ensure low-temperature strength and high-temperature ceramic bonds.

The total output of shaped refractories in industrial countries has progressively declined, while the output of monoliths (castables) has increased [1,5]. Currently methodologies for assessing environmental sustainability, such as product life cycle assessment, are often implemented too late in the design process [16–19] (Fig. 3). Integrating environmental sustainability modelling into computer-aided systems enables the evaluation of trade-offs between technical performance and environmental impact, thereby facilitating informed decision-making during the embodiment decision-making process [20]. Monolithic refractories (castables) are then produced via formulation design and manufacturing using materials jetting, additive manufacturing and 3D printing. The hypothesis is that computer-aided systems support the ability to explore and exploit cause-and-effect relationships across disciplines [20]. This facilitates the expansion of design elements while embedding sustainability-by-design principles at the design embodiment stage [20]. In this line, careful storage, preparation and integration of data and derived models will effectively facilitate management of cross-disciplinary non-linear relationships. This includes product conceptualisation (molecular dynamics, DFT, etc.). Sustainability-by-design principles offer a strategic approach to embedding environmental, social and economic sustainability into the design of monolithic refractories from the earliest stages. The approach aims to minimise resource use.

The main objective of geopolymer refractory castables is to create a matrix with good strength at room and high temperatures. The first hypothesis is that formulations based on the quaternary system: K_2O-(SiO_2)-Al_2O_3-H_2O, shorten as K-(S)-A-H, has advantages over CaO-Al_2O_3-H_2O, or C-A-H, with the expectation that the former are more thermally stable and provide better flowability.

3. Review and systematic analysis of the evolution in the sustainability of refractory materials

A bibliometric and analytical review was performed to understand how refractory materials have evolved in the context of climate change and the potential for global warming. Operating refractory materials in high-temperature processes (such as sintering) requires energy, which has an impact on global warming. Integrating the notion of a green future and sustainable development requires concepts such as cold

Table 1
Natural raw materials for cold setting refractory.

	Name	Description	Temperature of Decomposition and activation	Chemical components	Advantages
1	Kaolin	White refractory clay made of kaolinite, $Al_2Si_2O_5(OH)_4$	450-750 °C	SiO_2, Al_2O_3	The oxides transformed to mullite and cristobalite at high temperature
2	Bauxite	Red sedimentary rock made of aluminium oxide hydrate, $Al_2O_3.nH_2O$ with high fraction of iron minerals	200-750 °C	Al_2O_3	Insure refractoriness at high fraction
3	Talc	Silicate Magnesium Hydrate, $Mg_3Si_4O_{10}(OH)_2$	800-840 °C	MgO, SiO_2	Low thermal conductivity, high porosity, thermal shock resistance
4	Magnesite	Manesium carbonate, $MgCO_3$		MgO	Thermal shock resistance; low thermal conductivity

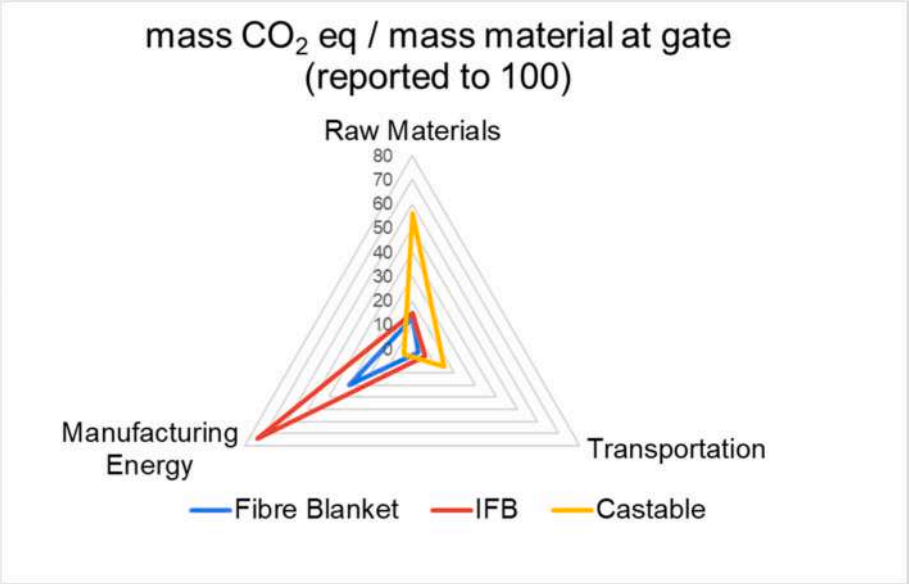


Fig. 3. Relative Climate change impact of various refractories as calculated by LCA (IFB = Industrial Fired Refractory Bricks) (data elaborated from Ref. [18]).

refractory materials, energy efficiency and sustainable raw materials in a circular economy. As the search space for validating the optimal sustainable process is very large, the review process and bibliometric investigations are combined to demonstrate the effectiveness of computer-aided, role model-based tools in ascertaining the cold process in the evolution of refractory materials into more sustainable, energy-efficient composites for refractory applications [21]. One opportunity is to use a computer-based framework that enables us to focus on databases and existing models associated with computational tools, in order to address challenges relating to the sustainability of refractory composites [22]. The following keywords were used to collect documents potentially related to the topic within the timeframe of 1923 to 2025: “cold refractory”, “thermal cycle”, “climate change”, “high temperature”, “clay”, “alumina”, “climate adaptation”, “sustainability”, “zero carbon”, “energy efficiency”, “resources efficiency”, “toxic emission”, “thermal process”, “colloid silica”, “nano materials”, “refractory products”, “steel making”, “dryers”, “furnace”, “Industry”, “artisanal”. As shown in Table 2, the results count 5459 documents that cite a total of 182,152 references retrieved from the Scopus database.

Fig. 4 shows the evolution of scientific production on cold refractory materials, including citations. It can be seen that there was very little scientific research on cold setting refractory materials before 1980, which is due to the fact that concerns about global warming did not arise until after this date. Concerns about the environmental impact of ceramics sintering (intensive energy use, CO_2 emissions, etc.) have led to an increase in scientific publications on cold refractory processes and technologies since the year 2000. By 2024, cold processes will be the primary method of refractory production. A significant increase in publications on cold-setting refractory was observed between 2015 and 2025. This exponential growth in scientific publications is correlated with the exponential growth in citations, demonstrating researchers' focus on more sustainable and environmentally friendly processes. The “Journal of the Cleaner Production” appears as the principal platform for the publications of the refractory materials with a h-index of 48 (Table 3). The traditional journals for ceramics publishing refractories, such as “Energy”, “Construction and Building Materials”, “Applied Energy” or “Ceramics International”, are no longer having high volume of scientific publications related to sintered refractories. The “Journal of the Cleaner Production” promotes green processes,

Energy efficiency and sustainability for the refractories of the future.

From the beginning of the history of refractory, the USA appears to be the leader, with 167 scientific contributions between 1920 and 1974. This figure grew to 2672 by 2009. Germany and Italy represent the largest volume of refractory materials in Europe, with their production increasing over time, reaching fewer than 100 scientific papers in 1985 [10]. By 2009, the two countries had published more than 900 scientific papers. China and India emerged as important producers of scientific papers related to refractory materials from 1990 to 1995. Today, they are among the five most important producers of sustainable refractories alongside the USA, Germany and Italy.

Table 2
Data on the evolution of cold setting refractory materials.

Description	Results
Timespan	1923:2025
Sources (Journals, Books, etc)	1983
Documents	5459
Annual Growth Rate %	3.16
Document Average Age	9.82
Average citations per doc	18.74
References	182152
DOCUMENT CONTENTS	
Keywords Plus (ID)	28112
Author's Keywords (DE)	11819
AUTHORS	
Authors	16135
Authors of single-authored docs	539
AUTHORS COLLABORATION	
Single-authored docs	603
Co-Authors per Doc	3.94
International co-authorships %	18.26
DOCUMENT TYPES	
article	3203
book	18
book chapter	201
conference paper	1541
conference review	106
data paper	2
editorial	4
erratum	1
letter	3
note	10
report	1
retracted	2
review	354
short survey	13

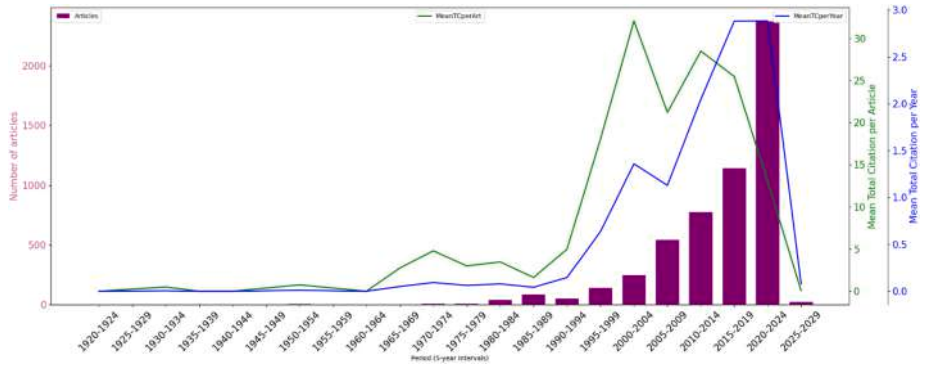


Fig. 4. Evolution of the scientific publications and citations of the terms correlated to the cold setting refractories.

Table 3
Classification of journals publishing articles on cold setting refractories according to h-index, total number of citations (TC), total number of publications (NP), and year of the first publications (PY_start).

Source	h_index	TC	NP	PY_start	Ranking on TC	Ranking on NP
JOURNAL OF CLEANER PRODUCTION	48	6722	137	2007	1	1
ENERGY	37	4450	75	1987	3	3
CONSTRUCTION AND BUILDING MATERIALS	36	5375	112	2009	2	2
APPLIED ENERGY	28	3617	35	1999	4	6
RESOURCES, CONSERVATION AND RECYCLING	20	2576	26	1994	5	8
APPLIED THERMAL ENGINEERING	19	1065	32	1997	6	7
FUEL	19	1046	36	2005	7	5
CERAMICS INTERNATIONAL	18	890	54	2005	8	4
ENERGY AND FUELS	17	799	26	2005	10	9
ENERGY CONVERSION AND MANAGEMENT	17	842	19	1997	9	10

4. Energy efficiency and design of cold setting refractories

4.1. Sustainable raw precursors for refractory castables

Kaolin, bauxite, talc, magnesite and so on have the advantage of retaining some degree of stability after dehydration or decarbonation, which guarantees the stability of refractory composites processed at low temperatures (see Table 1). Kaolin is a rock rich in kaolinite ($Al_2(Si_2O_5)(OH)_4$), a phyllosilicate containing aluminium hydrate that loses hydroxyl groups between 350 and 500 °C. This process is followed by the formation of metakaolin [23]. Metakaolin has high reactivity and can form thermally stable products at high temperatures. Further thermal transformation of metakaolinite produces spinel, mullite and other products with good refractory potential [15].

Bauxite is a rock found in tropical and subtropical climates. It is made up of iron and aluminium hydroxides and oxides, including Al$(OH)_3$. This material, known as the main ore of aluminium, includes the following minerals: boehmite, gibbsite and diaspore. Siderite, magnesite, quartz, hematite and goethite can also be found [23]. With an annual production of 110, 47 and 325 million tonnes respectively, Australia, China and Brazil are among the countries with the most abundant bauxite reserves in the world [24]. Dehydroxylation occurs during thermal treatment at temperatures between 200 and 750 °C. At 500 °C, gibbsite transforms to boehmite with a loss of intensity of the 3620 cm^{-1} and 3351 cm^{-1} OH stretching bands, which correspond to the deformation band at 1024 cm^{-1} (25. Dehydroxylation of gibbsite starts at 220 °C and ends at 350 °C, whereas for boehmite and diaspore it starts at 250 °C and ends at 622 °C and 650 °C, respectively. Reactive alumina is produced from bauxite via the Bayer extraction process and low-temperature calcination. Bauxite processing results in different oxides and hydrates that are useful for designing and producing cold-setting refractories.

Talc, $Mg_3Si_4O_{10}(OH)_2$, is a phyllosilicate not containing aluminium, member of the Pyrophyllite-Talc Group. Being a magnesium silicate hydrate, talc is decomposed to enstatite and amorphous silica between 800 and 840 °C [25]. Enstatite is transformed to clinoenstatite around 1200 °C and the amorphous silica changed to cristobalite. The thermal stability of the calcined product after 840 °C is significant for the use of this raw materials in the design and production of cordierite and mullite-cordierite like cold refractory composites.

Magnesite ($MgCO_3$) is an important raw material for refractories. It is a magnesium carbonate that decomposes at 350 °C to produce magnesium oxide (MgO), a very important oxide for the design and production of cold-setting refractories.

4.2. Aluminosilicate binders for refractory castables

The main components of traditional refractories are alumina and silica. It is well established that the transformation of natural kaolinite results in mullite ($3Al_2O_3.2SiO_2$), a high-grade refractory material. Advances in science and technology have led to new formulations that are energy efficient and environmentally friendly. In this class of materials, geopolymer castables are products that demonstrate excellent strength and densification at room temperature. However, glazing phases formed at relatively low temperatures in the initial formulations [26–28]. Adding Al_2O_3 and SiO_2 has been suggested as a way of improving thermal stability at high temperatures [15]. More recently, geopolymer castables have been proposed that use alumina, corundum and silica as aggregates [5,26–28]. Another alternative, recently identified by laboratory researchers, is to partially or completely replace the geopolymer binder with colloidal alumina or silica (3-Kamseu et al., 2009a). This significantly reduces the alkali's contribution to forming the glaze phase, which affects the thermal stability of refractory materials. This innovative approach optimises particle grading (Fig. 5). In detail, the particle packing and microstructure shown in Fig. 5 are ideal for the cold-setting refractory model proposed in this work. The model comprises coarse grains of refractory aggregates densified with small particles of an appropriate grade for optimum matrix densification. The matrix of aggregates uses an Al-rich binder with the capacity to ensure minimum mechanical strength before and after thermal cycles. The role of the

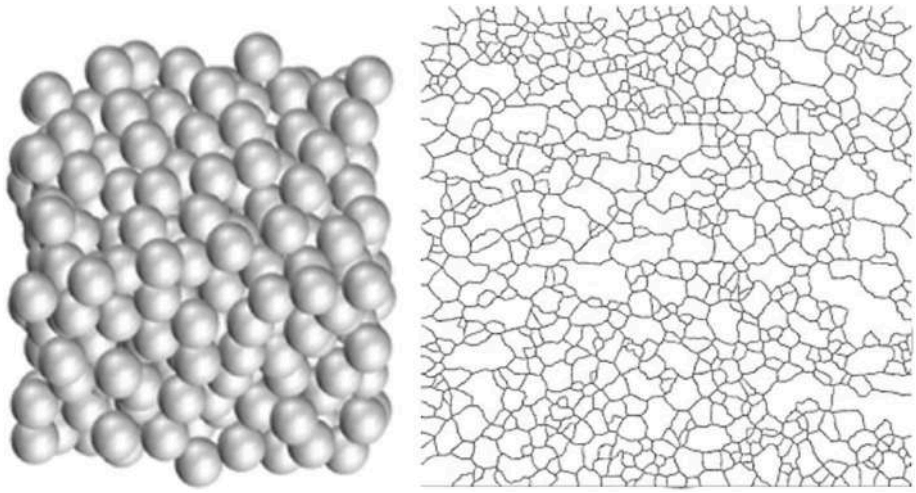


Fig. 5. Particles packing and the optimisation pattern of the shrinkage-free in cold-setting refractory composites [29].

aggregates is to ensure thermal stability and shrinkage-free transformations during heating and cooling.

Geopolymer gel chemistry involves the dissolution of alumina and silica precursors in an alkaline solution, resulting in the formation of inorganic monomers such as $Si(OH)_4$, $Al(OH)_4^-$, $SiO(OH)_3^-$ and $SiO_2(OH)_2^-$. These monomers play a significant role in achieving polycondensation of the gel and composites during the dissolution of the aluminosilicates. At high temperatures, nepheline, kalsilite, leucite and a few other phases crystallise [5,25]. These phases bond to the aggregates to form well-crystallized refractory composites (Figs. 6–8).

The crystallization or transformation of reactive precursors into crystalline phases (geopolymer-bonded refractories) is intrinsically linked to the phases developed and the processing route used. (i) the chemical composition, with control of the alkali content and optimisation of the colloidal phase; (ii) the solid/liquid ratio, with optimisation of the shrinkage-free system; and (iii) thermochemical reactions, in relation to the thermal treatment parameters used for castable refractories [5,25]. A key advantage of the geopolymer refractory binder is its ability to foster strong adhesion and interfacial bonds between aggregate particles, forming a ceramic matrix (see Figs. 9 and 10).

4.3. MgO-based aluminosilicate refractories

MgO-based materials, commonly known as periclase refractories, are widely used in the ferrous and non-ferrous metal industries due to their high melting point (2800 °C) and excellent resistance to basic slag corrosion [30]. MgO-based aluminosilicate refractories refer to cordierite-based ones. Cordierite, the most common form of which is present in high-MgO and high temperature refractory materials, offers high thermal and chemical stability, a low dielectric constant, a low thermal expansion coefficient and high mechanical strength, making it an appropriate material for thermal insulation. In the case of cold-setting refractory, the MgO-based binder is prepared as a cordierite-based geopolymer gel which transforms into cordierite upon heating, thereby promoting thermal stability at high temperatures. The development of MgO-, Al_2O_3- and SiO_2-based oligomers through geopolymer gels results in the formation of gels with amorphous content that crystallise at 900 °C. The metastable cordierite (μ-cordierite) formed at room temperature transforms into β-cordierite at 900 °C and into hexagonal cordierite at 1300 °C [31].

Geopolymerisation offers significant advantages in the low-temperature synthesis of refractory shapes and castables. The crystallization of α-cordierite begins with intermediate phases, such as spinel, sapphirine, cristobalite and mullite. The geopolymer gel remains amorphous even when thermal treatment is considered. Cordierite nucleates early in this process and remains stable throughout the temperature development, only changing its allotropic form [32]. Figs. 7 and 8 illustrate the thermal evolution of cordierite and mullite-cordierite-based geopolymer refractory composites. A large, diffuse diffraction peak centred around 28° 2θ [5] is characteristic of the

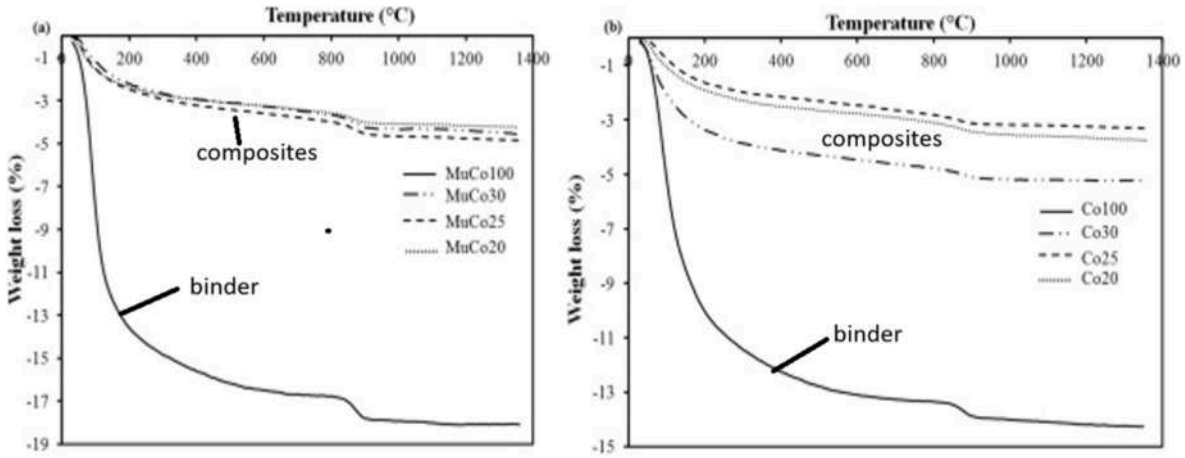


Fig. 6. Particles packing effects on the reduction of weight loss in the binder-aggregates composites (a) mullite-cordierite like and (b) cordierite-like refractory composites [5,25].

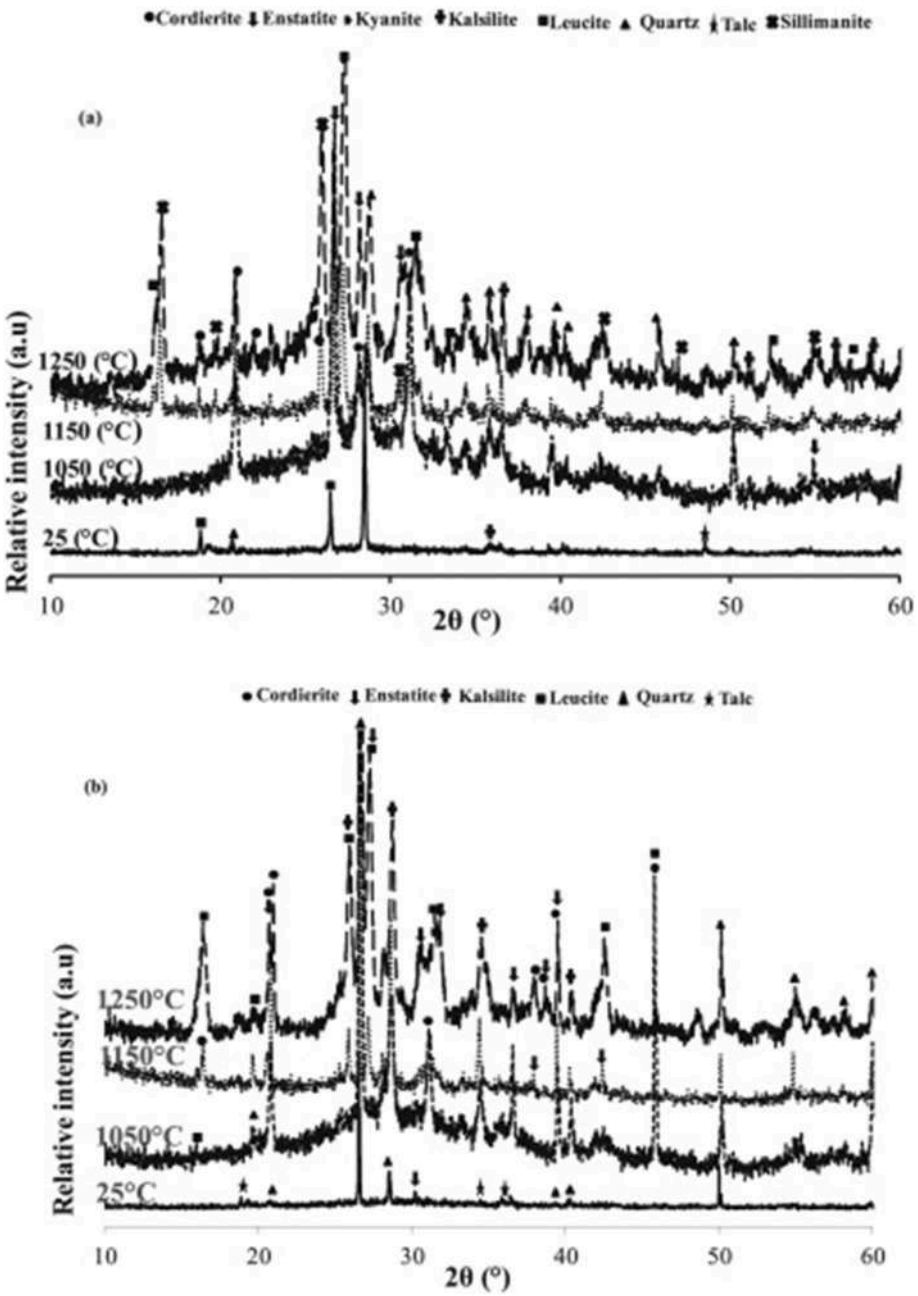


Fig. 7. Transformation of amorphous to crystalline phases during the sintering of cold setting mullite-cordierite (a) and cordierite-like refractory composites (b) Cordierite ($MgAl_4Si0_{18}$); Enstatite ($MgSiO_8$); Kalsilite ($KAlSiO_4$); Kyanite ($Al_2O_3{\cdot}SiO_2$); Leucite ($K(AlSi_2O_6)$); Quartz (SiO_2); Sillimanite ($Al_2O_3{\cdot}SiO_2$); Talc ($MgSiO_{14}(OH)_2$) [5,25].

amorphous structure of the geopolymer refractory gel [33]. During thermal cycling, peaks of cordierite, kalsilite, leucite and cristobalite accompany the amorphous phase, which progressively transforms into new crystalline phases. Above 1100 °C, the volume ratio of crystals increases significantly, as is the case with kyanite refractory aggregates [5, 25]. The transformation to mullite produces reactive silica, which forms secondary mullite in the presence of alumina in the gel [5,15]. Optimizing the content of aggregates allows the effects of dehydration, which take place between 100 and 700 °C, to be controlled and favours the formation of shrinkage-free matrices (Fig. 11).

5. Processing cold-setting refractories

One of the most popular applications of cold-setting refractory is refractory castables. These require a rheological process and grading of the grain size to optimise packing and ensure free shrinkage (Fig. 12).

5.1. Energy efficient calcination

The manufacture of chemically bonded refractory materials has demonstrated the significant role of reactive solid precursors such as metakaolin, hydrated alumina and fused silica [3]. These are described as binding accelerators. However, their production must be energy efficient with lower emissions to guarantee the overall sustainability of cold-setting refractory materials. Traditional coal- and fuel-powered kilns use heat supplied by the combustion of carbon-based fuels, which results in a significant proportion of emissions related to combustion, with the remainder due to chemical transformation. Substituting fossil fuel combustion with renewable energy calcination significantly reduces CO_2 emissions.

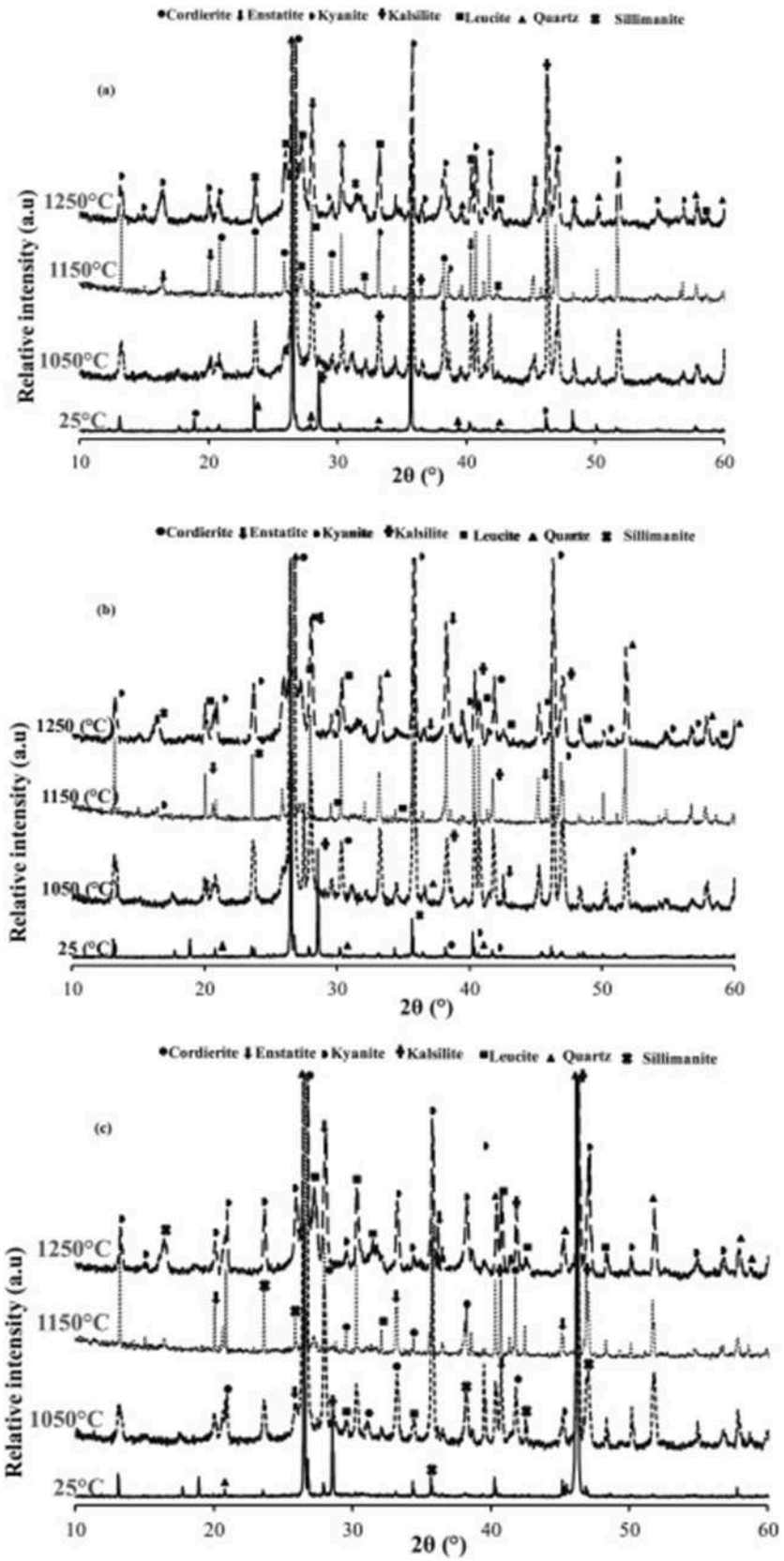


Fig. 8. XRD patterns of mixed refractories with cordierite (Co) and kyanite aggregates (Ky) (wt%): (a) 30 Co −70 Ky, (b) 25 Co - 75 Ky, (c) 20 Co - 80 Ky as a function of the temperature. (Cordierite ($MgAl_4Si0_{18}$); Enstatite ($MgSiO_8$); Kalsilite ($KAlSiO_4$); Kyanite ($Al_2O_3•SiO_2$); Leucite ($K(AlSi_2O_6)$); Quartz (SiO_2); Sillimanite ($Al_2O_3•SiO_2$)) [5,25].

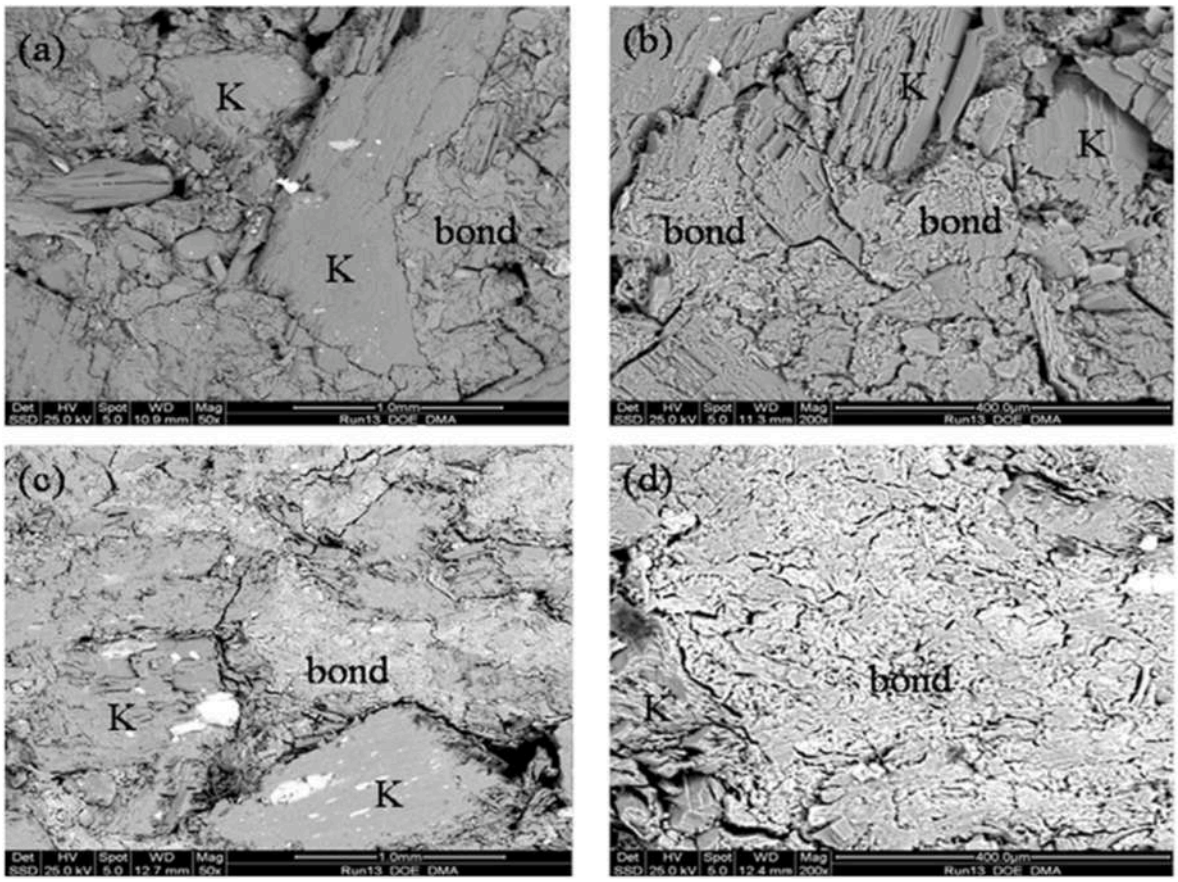


Fig. 9. Interparticles bonds developed into the matrix of cold setting refractory after crystallization. (a) morphology of the binder and grain of kyanite (K) (b) binder (c) grains boundaries (d) crystallized bond [5].

Recently, a range of innovative technologies have been developed and are being optimised for the production of reactive solid precursors (binding accelerators) [35,36]. These include microwave, thermal plasma, fluidised bed, solar, electric and thermoelectric with Joule heating technologies. The objective of this work is not to illustrate efforts to reduce global warming related to conventional kiln fuel or coal processing.

Microwave heating offers the potential to calcine materials without producing toxic emissions. Heating the bi-polar hydroxyl groups results in dehydroxylation. Heat is generated inside the material itself, so there is no need to use burners. There is no waste heat, so there is no need for refractories in the heating chamber except for the transport belt. There are also no exhaust gas emissions.

The microwave system uses electricity in suitable areas. Self-generation of electricity can be considered to significantly reduce production costs and avoid the complications of handling fuel. Precise temperature and heat control can efficiently monitor the calcination process and lower specific energy consumption. This technology has already been used on an industrial scale, as well as in a hybrid solution combining microwaves and a rotary kiln. The initial energy requirements evaluation shows values between 2.35 and 3.5 kJ/g of calcined clay [37]. However, challenges remain regarding the extent to which microwave radiation penetrates the volume of solid precursors and the decrease in resistivity after dehydroxylation, which can induce recrystallization.

Thermal plasma processing for the calcination of clays considers a range of methods involving the exposure of the raw solid precursor to ionised gas [37]. Ionised gas is highly conductive and allows current to pass in the form of an electric spark. This allows collisions between electrons and larger particles to become more frequent, transferring the electron's kinetic energy. The recombination of charged particles increases the temperature of the gas [37,38]. Apart from calcination and sintering, thermal plasma technology is also used for waste processing (incineration). Although thermal plasma processing presents high potential for energy-efficient calcination of clays, very high temperatures can be problematic for suitable dehydroxylation of clays [39–41].

Fluidised bed technology refers to a system in which gas passes through a bed of solid precursor particles that are relatively fine [42–44]. When the gas flow rate is high, the particles become suspended in the gas flowing upwards. The advantages of fluidised beds include saving fuel (energy), due to the high heat transfer coefficient and high contact potential between the hot gases and clay particles [43,44]. There is also more homogeneity in the products of calcination. However, industrial scaling still requires serious study to make this process economical and available on the open market.

Solar thermal processing is the most promising method for transforming hydrated aluminosilicates into reactive solid precursors (binding accelerators). Concentrated solar thermal energy (CSTE) plants can now reach temperatures of 550–1150 °C, which are suitable for the calcination of kaolin and alumina hydrates as bauxite [45]. A rotary kiln furnace, similar to those used for calcination powered by fossil fuels, is heated using concentrated solar radiation. Davis et al. [46] described scaling up the thermochemical calcination of alumina using concentrated solar thermal radiation in a solar reactor, eliminating the need for combustion input and associated CO_2 emissions. However, the authors evidenced the need to improve the extent of calcination and energy efficiency as the process is scaled up. It is in fact expected that large quantities of alumina hydrates would lower reactor temperatures due to longer particle residence times and the relatively lower heat losses associated with large-scale reactors. X-ray diffraction analysis revealed a

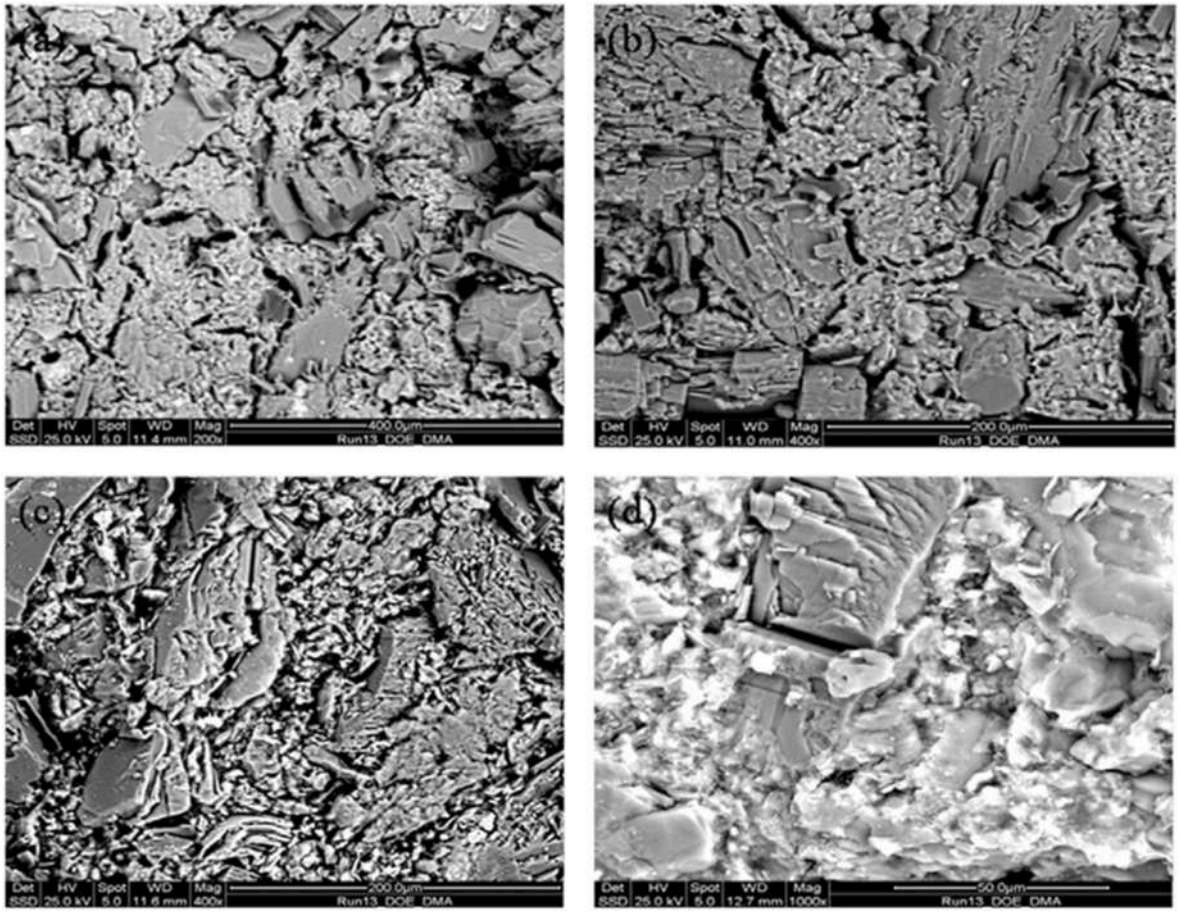


Fig. 10. Morphology of the cold setting refractory matrices: (a) efficiency of the crystallization of geopolymer bond during heating cycle (b) morphology of grains (c) grains expansion during heating (d) descriptive grains boundaries after crystallization [25].

high γ-Al_2O_3 phase fraction and a low α-Al_2O_3 phase fraction in the concentrated solar thermal processing product. Pasabeyoglu et al. [45] presented the transformation of kaolin to metakaolin using a solar reactor in a rotary kiln and a solar simulator [47]. The transformation of kaolin to metakaolin through CSTE does not emit CO_2 as fuels and coals are not combusted (Fig. 13).

5.2. Processing the solid precursors and binders

Grading of particles that yields maximum solid density and maximum interlocking of particles is appropriate for a free-shrinkage castable refractory, where the binder (in the form of nano- or meso-particles) occupies the voids resulting from densification of the aggregates to form one compact block. The raw materials need to be organised into various grades to enable better packing in a dense system. First, pack the coarse particles as densely as possible, maintaining the initial configuration. Then, fill the voids with medium particles. The fines aggregate then follow without disturbing the packing of the coarse and medium particles. The fines occupy the voids between the coarse and medium particles, and some of them could be similar in size to the binders, in order to minimise shrinkage potential of the binder [29,49]. The dense matrix of the cold-setting refractory made from different particle classes with maximum particle interlocking allows the reduction of the required water and improves thermal shock resistance [29, 49].

Hydratable alumina, colloidal alumina, colloidal silica and micro-silica, etc., are innovations resulting from advancements in the science and technology of cold-setting refractories [24,26,50]. The main role of nanomaterials is to improve thermomechanical properties while reducing energy consumption and improving densification at low temperatures. The positive effects of particle grading and nanomaterials in cold setting refractory processes are twofold: (i) an exponential increase in the proportion of atoms at grain boundaries, and (ii) the generation of dislocations, quantum confinement, etc., in the microstructure. This contributes to improvements in compressive and tensile strength, ductility, resistance to thermal shock, abrasion and chemical corrosion, aggregate-paste bonding, crack control and self-healing. The bonding mode of refractory castables shifts from polymerisation to coagulation. In the polymerisation process (in the case of geopolymers), the refractory aggregates and binder are embedded in a structure in which the ceramic bonding process starts at a relatively high temperature. Innovative binders are prepared from an alkali-silicate colloidal solution or colloidal alumina or silica for packing particles of aggregates designed to withstand thermal shock, spalling potential and shrinkage [5]. The robust hot strength of such matrices ensures this new class of refractory has exceptional thermo-mechanical characteristics and is environmentally sustainable [5–25,51].

5.3. Manufacturing process

Significant progress has been made in the sustainable preparation of raw materials for cold-setting refractory binders and composites (Table 4). These works include the thermochemical transformation of raw aluminosilicates into reactive precursors (drying and calcination) and particle packing to enhance densification. As a result, the manufacturing process has become as simple as referring to the requirements of the final products in terms of densification, porosity, shape, etc. Of the conventional ceramic forming techniques, such as slip

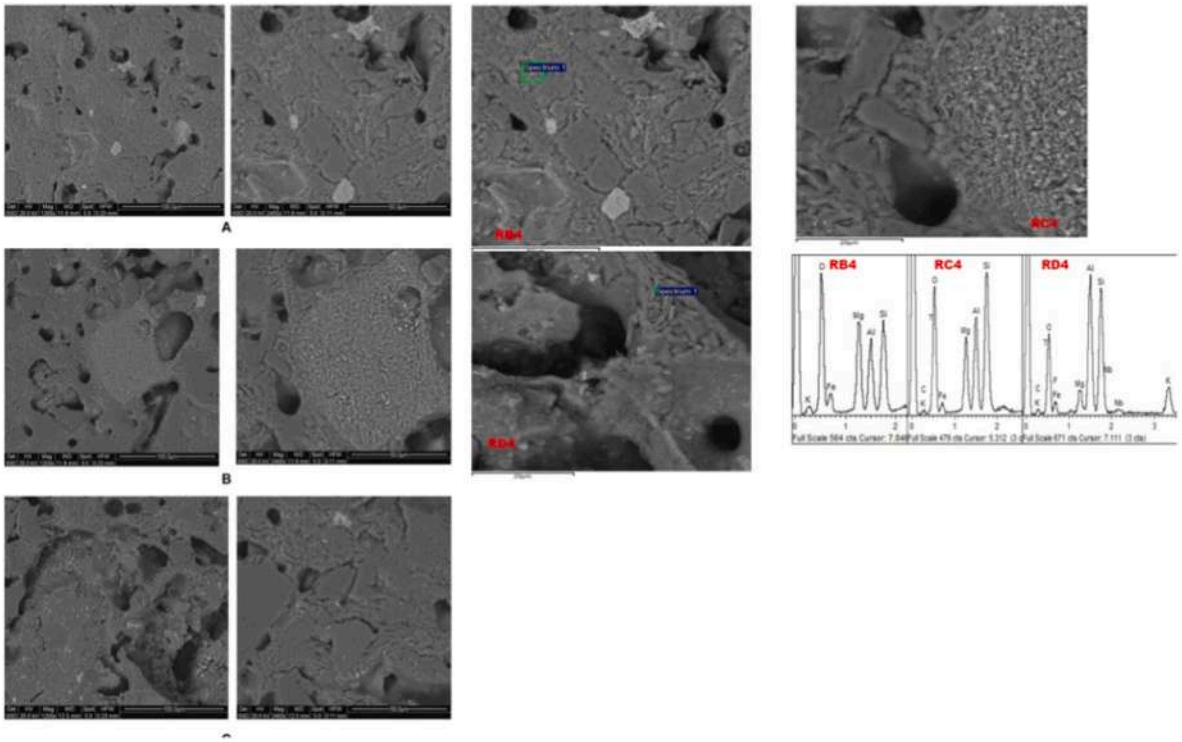


Fig. 11. Selected micrographs of cold-setting refractory matrix showing the effects of corundum addition into the thermal resistance of matrix upon firing cycles (samples A, B and C) and the nucleation and crystallization of Al-rich grains according to the glass content [34].

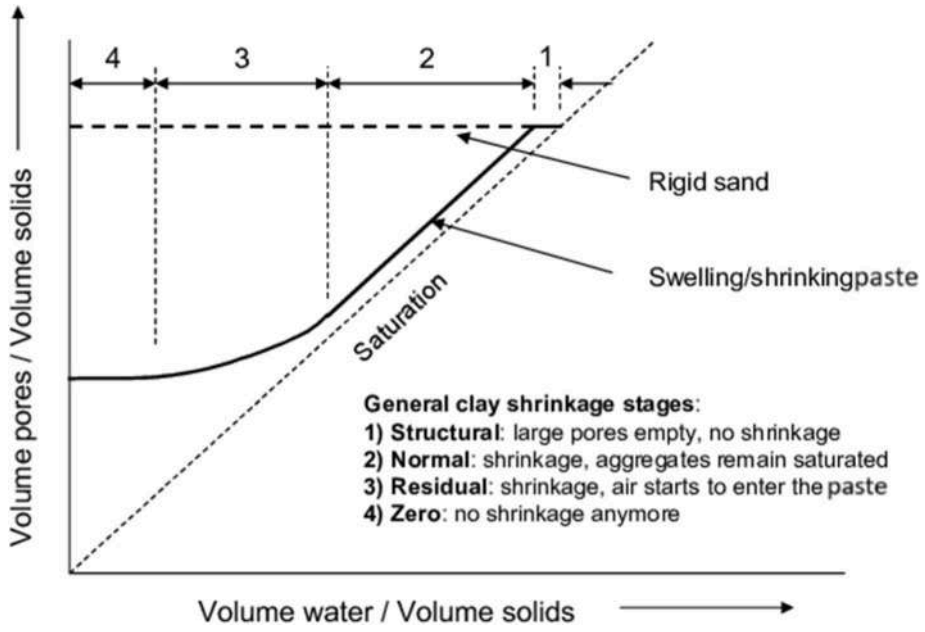
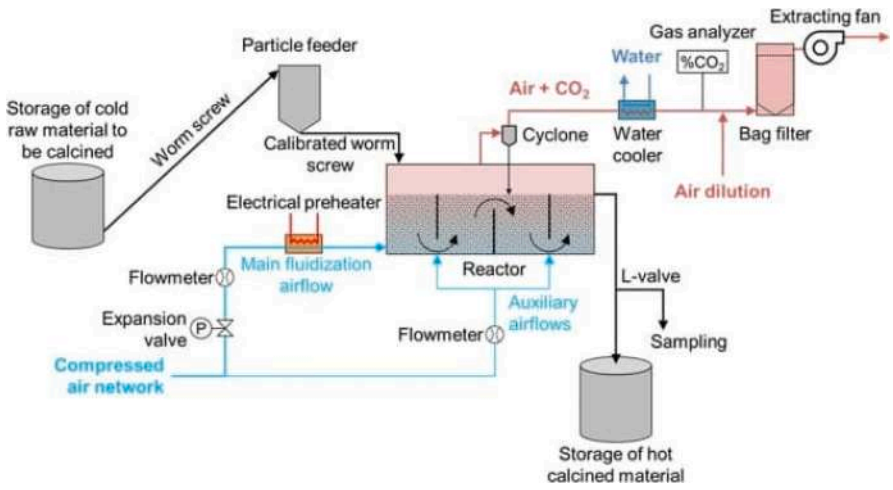


Fig. 12. Descriptive mechanism of shrinkage-free in the process of the densification of cold setting refractory applying particles packing theory [29].

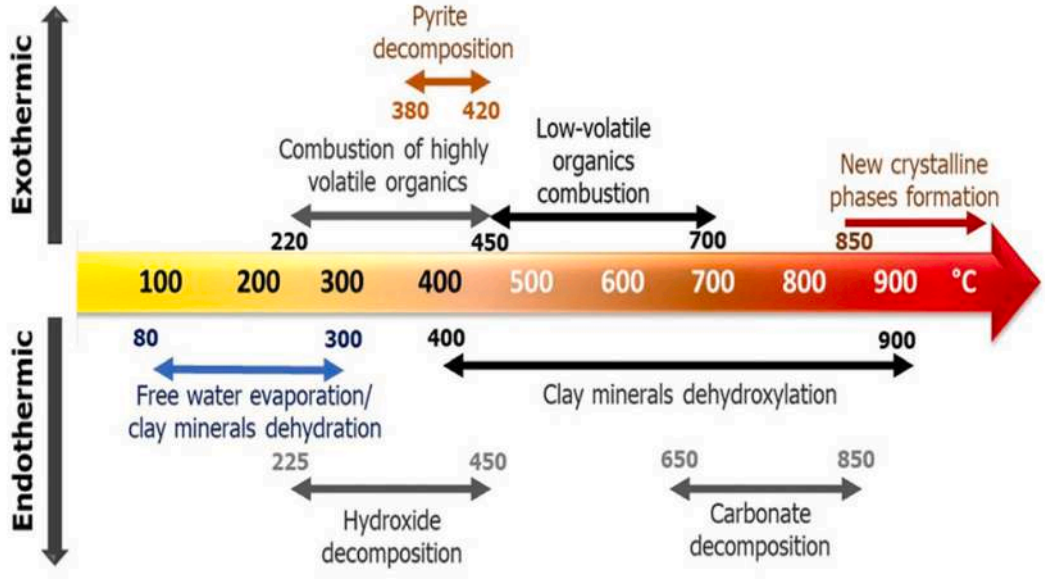
casting, extrusion, moulding and pressing, as well as the innovative additive manufacturing or improvement of pressing methods, two manufacturing methods have emerged as significant for the production of cold-setting refractories: the first is cyclic isostatic pressing and the second is direct ink writing [52].

Of the various additive manufacturing methods, direct ink writing (DIW) is a particularly straightforward approach that allows for the creation of products with intricate structures by depositing layers of extruded slurry [52]. DIW is a unique technique that introduces design freedom, multifunctionality and stability into its printed structures simultaneously.

DIW is an extrusion-based AM technique that can be used to fabricate 3D structures with complex architectures and compositions at the mesoscale and microscale. During the process, viscoelastic ink is extruded layer by layer through a deposition nozzle onto a computer-controlled translational stage to build up scaffolds and other 3D geometries. DIW has emerged as an innovative approach in relation to the various methods that have been developed to manufacture lightweight



a)



b)

Fig. 13. (a) Kaolin calcination using solar energy; (b) Temperature range of typical reactions taking place during the calcination of clays (figure elaborated from Ref. [48]).

Table 4
Evolution of the alumina-silicates refractory.

Period	Description	Problems solved	Technology	Observations/references
1820	Crushed rocks; silica bricks	First design processes of refractory	Conventional sintering	Traditional refractories
1880	MgO based bricks	Need of basic refractory		Technology enhanced processes
1902	Mullite-Alumina-zirconia Fused Alumina electrocasting			
1934	Tabular alumina	Rapid sintering		
1940-1950	Castables: in situ mixing CAC		Calcium alumina based castables	High technology and process for ultra-high performance
1951	Tabular and fused alumina aggregates for refractory concretes	Dry mix refractory concretes		
1960-1980	Cement-aggregates based refractory concretes: particles packing	Reducing binder and optimizing densification		
Since 1990s	Monolithic	Low-cement castables Self-flow refractory systems	Low-cement based castables	Technology and process driving by the Global Warming and Sustainable future preoccupation
Context of climate change	Energy efficiency and low emissions monolithic	Energy and CO_2 emissions	Colloidal mixes and pressing	

mullite insulation refractory, including foam-gel casting [53], pyrolysis of organic additives [54] and template replica [55]. However, it has proven challenging to achieve hierarchical pore structures and complex geometries using these methods. Additive manufacturing is an increasingly popular technique for applications requiring complex geometries [56]. It is currently being widely used to produce porous ceramics and dense refractory materials [57,58]. Direct ink writing stands out among the various additive manufacturing methods as a straightforward approach, enabling the production of intricate structures by depositing layers of extruded slurry [52]. Lightweight mullite insulation refractory has gained widespread use in the metal smelting and energy industries due to its exceptional high-temperature strength, light weight and superb thermal insulation properties.

Cold Isostatic Pressing, CIP, is a well-known moulding technique in which flexible moulds filled with powder are placed under equal, multidirectional pressure at room temperature, with moisture acting as the pressure-transmitting medium. In the case of cold-setting refractory composites, high uniformity of density is achieved. According to the bulk composition of the raw pastes, forging or hot isostatic pressing is performed [52]. This forming technology can be used to create specimens with complex shapes and structures. Cold Isostatic Pressing is classified as wet bag or dry bag depending on the powder loading mode and compression form. CIP is one of the most widely used forming techniques for producing ceramic products with high length-to-diameter ratios or complicated shapes [53]. During the wet bag process of cold isostatic pressing, powders or granules are filled into an elastic mould, which is often vacuum sealed later on. The filled mould is then placed in a fluid, and applying high pressure to the fluid achieves very homogeneous compaction of the ceramic body [54]. Unlike uniaxial pressing, cold isostatic pressing produces much lower density gradients, resulting in greater mechanical reliability of ceramic products [55,56]. Several authors have studied the cyclic uniaxial and isostatic compaction of ceramic, metal, and composite powders. Cycling at maximum pressure in particular resulted in smaller density gradients and stronger densification ([57]; [59]). For single-phase powders, considerably higher densification was observed with cyclic pressing. This was explained by improved particle packing, a higher particle coordination number, and the fracture of agglomerates [57,59,60]. For composites, an even greater relative improvement in densification was attributed to volumetric mismatch between different phases during pressure changes [52, 60–63]. In general, cyclic cold isostatic pressing has only been investigated for fine-grained ceramics. However, the application of this technique to coarse-grained oxide ceramics, such as refractories, remains unexplored.

6. Discussion

Refractory materials for a green future should be produced using cold setting processes such as geopolymerization, rather than sintering. Refractory matrices must have a long life cycle and must not emit toxic gases. The sustainable processes described in this review promote 'green' composite matrices, including bio- and eco-refractories. These are made from renewable materials, such as clay and alumina hydrates, as well as recycled industrial wastes suitable for refractory purposes. Natural raw materials are calcined at low temperatures using energy-efficient methods, such as solar thermal processing. The solid precursors obtained are processed by packing the particles and manufacturing them using isostatic pressing, 3D printing, or inkjet printing. Preparing colloids is fundamental, as is balancing the alkali content of the binder to ensure its efficiency and the extent of refractoriness.

The role of digital technologies at the intersection of decarbonising the production process of refractories (including improving energy efficiency) and meeting future development needs is in line with the necessity to optimise this field, which lies at the heart of industrial development. Molecular dynamics and DFT are vital for designing cold-setting refractory matrices that can withstand shrinkage, high temperatures and micro-cracking. Correlations between formulation, properties, and performance play a significant role in modelling, predicting, and simulating high-performance refractories that offer advantages such as low energy requirements for production, decarbonisation, and sustainability. Available literature shows significant progress in the development of cold-setting refractories. However, the challenge remains to develop an effective binder that can withstand both low and high temperatures. Geopolymerization using an alkaline solution in which an important fraction of the alkali is replaced by colloidal silica or alumina is thus a promising approach.

7. Conclusion

This review aimed to present the evolution of processes used in aluminosilicate refractory production, highlighting the impact of climate change and the need for sustainable development. The following conclusions are drawn from the results.

- Researchers and industries are working effectively to transition from sintering to cold-setting processes in the production of refractory materials, achieving energy efficiency and sustainability through innovative technologies.
- The cold-setting processes reduce the need for energy, toxic emissions and temperature, and significantly reduce costs.
- The particle processing and mix design allow for the production of refractory materials that can be used through pressing, moulding or 3D printing, with the ability to control the shape and achieve shrinkage-free matrices.
- The decarbonisation of refractory production benefits from innovative technologies that use machine learning and deep learning to optimise processes from raw material processing to the final product. This involves correlating formulations, properties and performance to better predict the behaviour of cold-setting refractories in service and their long-term durability.
- Cold-setting refractory materials are still posing a challenge to the potential vitrification of alkali-based crystalline phases at high temperatures.

CRediT authorship contribution statement

Elie Kamseu: Writing – original draft, Methodology, Formal analysis, Data curation, Conceptualization. **Paolo Veronesi:** Writing – review & editing, Methodology, Funding acquisition. **Lauraine Tiogning K. Djiogue:** Writing – original draft, Methodology, Formal analysis. **Joseph Marae Djouda:** Writing – original draft, Formal analysis, Data curation. **Isabella Lancellotti:** Writing – review & editing, Methodology, Data curation. **Elena Colombini:** Writing – review & editing, Writing – original draft, Formal analysis, Data curation. **Cristina Leonelli:** Writing – review & editing.

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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.

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