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Mechanical activation of expired Portland cements: Experimental characterisation

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ABSTRACT

This study examines the mechanical recycling of expired Portland cement through fine grinding at four different fineness levels: 390, 450, 550, and 650 m²/kg. To evaluate performance recovery, experimental tests such as compressive strength, calorimetric analysis, and setting time were carried out. The findings indicated that mechanical characteristics and reactivity were improved by increasing fineness. Cumulative heat release increased from 135 to 290 J/g, and compressive strength increased from 15 MPa (REC 390) to 36 MPa (REC 650). Higher fineness did, however, also result in a greater need for energy. REC 550 was determined to be the best formulation by applying the Technique for Order Preference by Similarity to Ideal Solution (TOPSIS) decision-making approach with equal weighting, which provides a good balance between energy efficiency and performance. The results support the possibility of recovering expired cement to create useful binders, which would help with waste minimization and environmentally friendly building.

Keywords: Expired cement; Mechanical activation; Calorimetry; Fineness; TOPSIS.

1. INTRODUCTION

In the last few years, the construction industry has taken a positive direction, based on the expected sustainable development manners, to reduce environmental pollution through the establishment of several environmental regulations and international agreements. Numerous studies have been carried out to minimise construction waste and greenhouse gas emissions in this social context [1]. The cement production is responsible of the world's CO_2 emissions for 5 to 10% [2,3]. Decarbonation of $CaCO_3$ in the raw material during the clinkerization process accounts for 65% of the CO_2 generated during cement manufacturing. with the remaining 35% coming from fuel combustion [1,2].

Several studies have been carried out to reduce construction waste [3–6]. Globally, around the world, more than 4 billion tonnes of waste are generated per year and could increase by nearly 200% by 2050 [7]. An important part of Algeria's economy is the construction industry, where local cement market is anticipated to continue growing, reaching over 46 million tons by 2025 [8]. However, following the epidemic of the coronavirus, the government of Algeria proclaimed the cessation of construction sites on 12 March 2020 in order to limit the spread of the virus. This decision had a significant impact on the public and construction works sector, as the health crisis disrupted construction projects in progress [9], the halt in construction activity also led to the depletion of cement stocks, with problems such as torn bags, prolonged storage and humidity contributing to the deterioration of cement [10]. The formation of portlandite and ettringite begins with the appearance of the Calcium Silicate Hydrate (C-S-H) gel after only one week of unfortunate exposure of the cement; three different patterns were observed; free lime absorbs the water when the relative humidity is between 10 and 14%, depending on the crystalline structure of the cementitious minerals [11]. Correspondingly, tricalcium aluminate (C_3A) reacts at a relative humidity of 80%. while tricalcium silicate(C_3S) and dicalcium silicate (C_2S) react at around 63% and 64% relative humidity, respectively [12]. This can delay the setting of cements exposed to moisture [13].

Reactivating expired cement (REC) uses a variety of techniques to recover its mechanical qualities and usability, which lowers waste and its negative effects on the environment. These techniques include chemical,

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mechanical, and thermal procedures; each has unique advantages and workings. By reducing the need for fresh raw materials, reactivating expired cement not only supports sustainable building methods but also the ideas of the circular economy [14]; the main techniques for reactivating EC are listed below:

- **Thermal reactivation:** by heating pre-hydrated (PH) cement powder to temperatures between 400 and 800 °C, it is possible to reactivate expired cement. This process uses less energy and produces less CO_2 than traditional cement production. The ideal temperature for reactivation, especially for Ordinary Portland Cement, is approximately 600 °C, which yields mechanical strength values comparable to those of new cement [15,16].
- **Chemical reactivation:** alkali metal hydroxides and salts like NaOH and $MgCl_2$ are common activators that improve the cementing qualities of EC by encouraging hydration processes [17]. Moreover when combined with EC, additives such as triethanolamine (TEA) and triisopropanolamine (TIPA) have been demonstrated to boost compressive strength; TEA alone has been proven to increase strength by 8% [18–20].
- **Recycling and regeneration:** It is possible to use EC as a mineral component in new cement compositions, replacing up to 35% of the cement's weight. By reusing EC that still meets strength standards and chemical requirements, it is possible to improve sustainability without compromising performance [10,21,22]. On the other hand, incorporating mineral additions such as fly ash (FA), silica fume (SF), ceramic, and glass waste powders into the EC matrix can greatly improve its compressive and tensile strength [23–27].
- **Mechanical reactivation:** involves subjecting inactive cement particles to mechanical beats with a pin mill to reduce their size to their initial fineness and release non-hydrated layers. Reusable unhydrated particles are revealed by grinding hydrated cement conglomerates, which enables them to resume their role as a cementitious binder. This technique improves the reusability of cement components, while eliminates the need for heat treatment, making it both sustainable and energy-efficient [28]. Following the pre-hydration phase, it is essential to adequately grind the cement to facilitate the hydration reaction, as it loses the fineness specified in the technical data sheet. The speed of the initial reaction between cement and water is directly correlated with the specific surface area of the cement; therefore, the grinding process is meticulously monitored to achieve a product with the desired fineness attributes: the intended percentage passing through a 45 µm sieve and the specific surface area [29].

Prehydrated cement mechanical activation has drawn a lot of interest as the suitable activation method to increase cement's hydration reactivity. As it increases surface area to enhance hydration without the need for high temperatures or chemical additions, it is a highly desirable technology that works technically, economically, and environmentally. It provides a sustainable and affordable alternative to conventional approaches.

The impact of mechanical activation on the hydration and strength development of Portland cement was investigated by SOBOLEV *et al*. [11] utilizing vibro-milling, they systematically varied the fraction of activated cement replacement and the milling time, mechanical activation enhanced compressive strength and early-age structure development. The advantages of improved hydration were specifically demonstrated by an improvement in compressive strength after one day and after 28 days. Analysis using scanning electron microscopy and X-ray diffraction (XRD) verified that the finer and more densely packed activated cement particles resulted in better hydration kinetics [15].

SANYTSKY *et al*. [30] also used a vibro-milling approach to increase the surface area and reactivity of Portland cement in order to investigate the effects of mechanical activation on its characteristics, the surface area increased to 510 m^2/kg after 60 minutes of activity and to 640 m^2/kg after 90 minutes. In comparison with ordinary Portland cement (OPC), this activation greatly increased the heat of hydration, resulting in a 14.4% higher heat release at 24 hours. Additionally, the activation shortened the initial setting time for the activated cement from 150 minutes for OPC to only 40 minutes. By reaching 59.6 MPa after 28 days, the modified cement demonstrated fast early strength development and satisfied the specifications for high-strength binders. Using a rotary high-speed mixer, BARABASH *et al*. [31] investigated the effects of mechanical activation on cement-water compositions and solutions. They found that a number of characteristics are considerably impacted by mechanical activation, in particular, it increases the exothermic heating of the hardening component and accelerates cement hydration. Additionally, the study found that a major factor in cement mixture workability is mechanical activation.

Additionally, GORBOVOY *et al*. [32] focused their study on how cement-water compositions are affected by mechanical activation. Their research showed that the water-solid ratio may be effectively decreased from 0.42 (without activation) to 0.38 (with 180 seconds of activation) by mechanically and chemically activating cement-water compositions, the hydration processes are accelerated by mechanical action, the authors added, the enhancement of the exothermic heating of the cement-mixing components confirms this acceleration to improve cement characteristics during hydration.

Although several studies have been conducted to investigate the mechanical and chemical activation of cementitious pastes and their incorporation as supplementary cementitious materials [33–38], as well as the influence of particle size on hydration kinetics and mechanical performance [39,40], the influence of fineness on the potential reactivation of expired Portland cement has not been thoroughly examined. This study aims to assess the reactivation efficiency of pre-hydrated cements subjected to varying grinding levels of fineness. The evaluation is conducted using a comprehensive multi-technique approach that includes thermogravimetric characterization, hydration kinetics measurement, compressive strength testing, and XRD-based crystallographic and chemical phase analysis on standardized mortars prepared with reactivated expired cement. Subsequently, an optimization analysis is performed to ascertain the ideal fineness by linking performance metrics with energy consumption, adhering to circular economy principles, using the Technique for Order Preference by Similarity to Ideal Solution (TOPSIS) multi-criteria decision-making framework.

The TOPSIS method has been shown to be an efficient and impartial multi-criteria decision-making (MCDM) tool for methodically assessing and ranking such alternatives. Prior research SWATHI and VIDJEAPRIYA [41,42] has confirmed its use in cementitious systems, hence supporting its incorporation into material selection and optimization procedures.

The primary goal of this study is to investigate the possibility of recycling expired cements, thorough the examination of their mineralogical, chemical, thermal and physical characteristics, in order to track hydration kinetics; the research uses calorimetric studies to analyze the heat of hydration, as well as XRD analysis for the detection of the different phases present before and after prehydration. Consistency tests to determine the amount of water needed for cement particle hydration, and an evaluation of the mechanical strength of the recycled cements at various fineness levels (390, 450, 550, and 650 m^2/kg) both in the short and long term. Additionally, it makes it possible to determine the best mechanical reactivation cycle that produces balanced performance with properties similar to OPC using TOPSIS method.

2. MATERIALS

The materials used in this study consist of Ordinary Portland Cement (OPC) and Recycled Expired Portland Cement (REC), as shown in Figure 1. Density and specific surface area were measured following the NF P15-435 [43] and NF EN 196-6 [44] standard respectively. The REC used in this study was obtained from local construction stock and had been stored for about seven months starting in March, beyond its expiration date, under

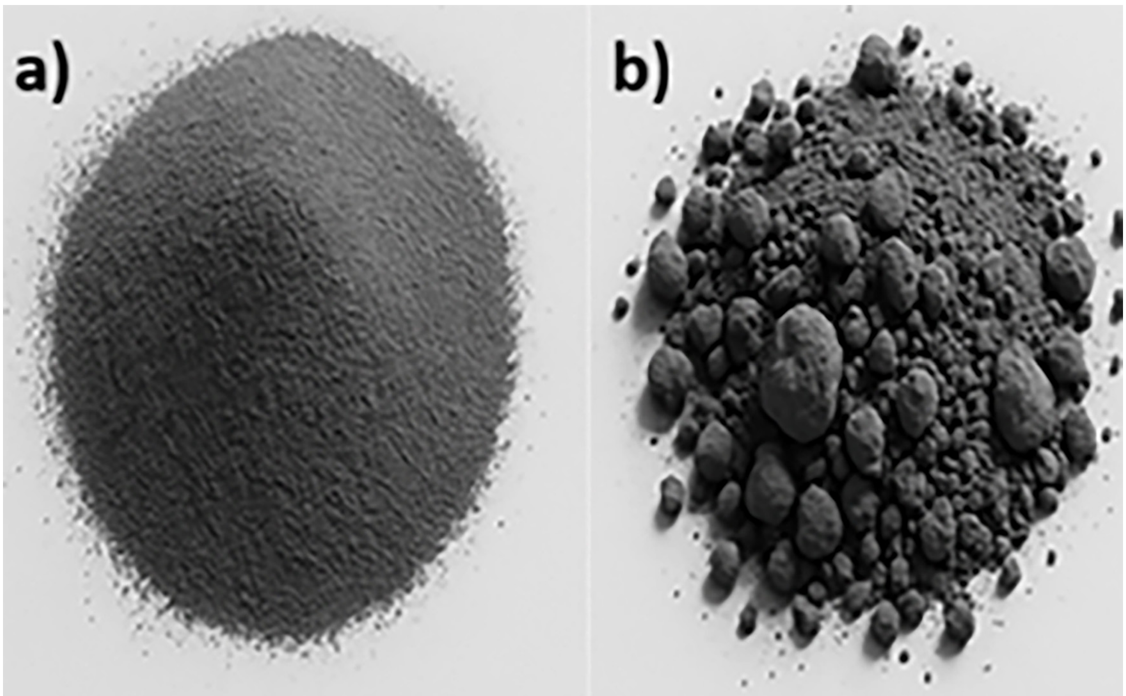

Figure 1: (a) OPC, (b) REC cements.

Table 1: Chemical composition from x-ray fluorescence analysis [52].

CHEMICAL COMPOSITION %	CaO	SiO_2	Al_2O_3	Fe_2O_3	SO_3	MgO	Na_2O	K_2O
OPC	62.61	19.61	4.37	2.85	2.57	2.83	0.20	0.71
REC	62.60	19.04	4.29	2.85	2.37	2.54	0.20	0.71

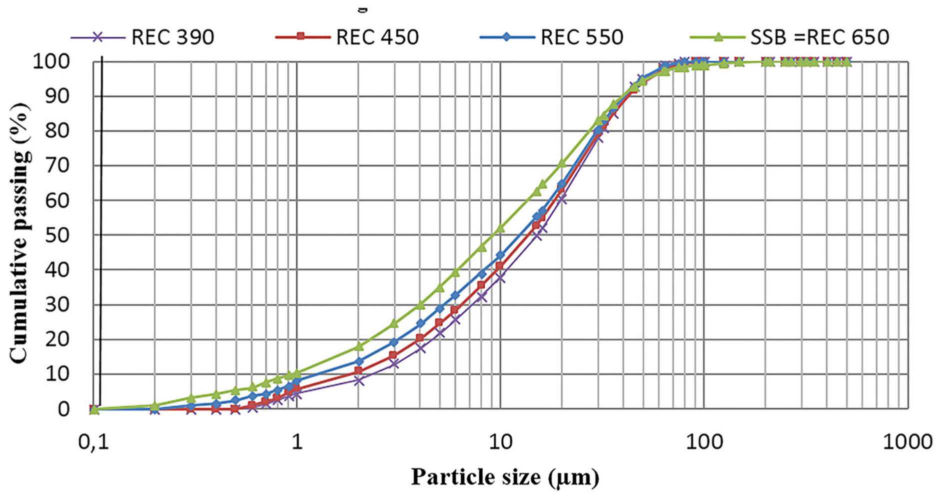

Figure 2: Particle size distribution of OPC, and REC using laser diffractometer.

uncontrolled ambient conditions. The intentional exposure of the cement to these uncontrolled environmental factors was designed to replicate real-world scenarios in which cement bags are kept under shelters but remain in direct contact with ambient climatic conditions. This approach realistically simulates the situation commonly encountered in the construction sector in Algeria following the sudden interruption of activities and the nation-wide lockdown measures imposed during the coronavirus epidemic.

The chemical and physical properties of the REC and OPC cements are summarized in Table 1, while the particle size distributions of the REC cement are illustrated in Figure 2.

Standard sand is used in this study according to EN 196-1 [46], to estimate the cement recycling effect.

3. EXPERIMENTAL METHODS

In this investigation, OPC is utilized in its natural state, without any drying or pre-treatment. Conversely, REC is initially present as hardened agglomerates, is subjected to a grinding in a cylindrical ball mill after a drying process at 105° to attain varying degrees of fineness as illustrated in Figure 3. By varying the grinding time as seen in Table 2, four distinct particle size distributions are obtained (Figure 2), corresponding to the samples REC 390, REC 450, REC 550, and REC 650, which will be the focus of this research.

3.1. XRD analysis

The identification of crystalline phases in the OPC and EC samples was conducted using XRD with a **PANalytical Empyrean** diffractometer. The device is equipped with a Cu Kα radiation source (λ = 1.5406 Å), operating at a voltage of 45 kV and a current of 40 mA. The present system utilises the Bragg-Brentano reflection geometry (2θ), a configuration that is particularly well-suited to powder analysis.

OPC and EC differ mineralogically, according to the XRD examination (Figure 4). A large concentration of unreacted clinker phases is indicated by the strong, sharp peaks for alite (C_3S) and belite (C_2S) in the OPC pattern. The EC sample, on the other hand, shows lower intensities for these phases along with sparse indications of calcite ($CaCO_3$) and portlandite ($Ca(OH)_2$), indicating partial carbonation and hydration. These modifications demonstrate that prehydration causes a reduction in reactivity, which might negatively affect the cement's functionality in practical applications.

3.2. Thermals analysis

Thermogravimetric analysis was performed using a **Setaram LABSYS evo S60** thermal analyser. The present apparatus facilitates the execution of simultaneous thermogravimetric analysis (TGA) and differential thermal

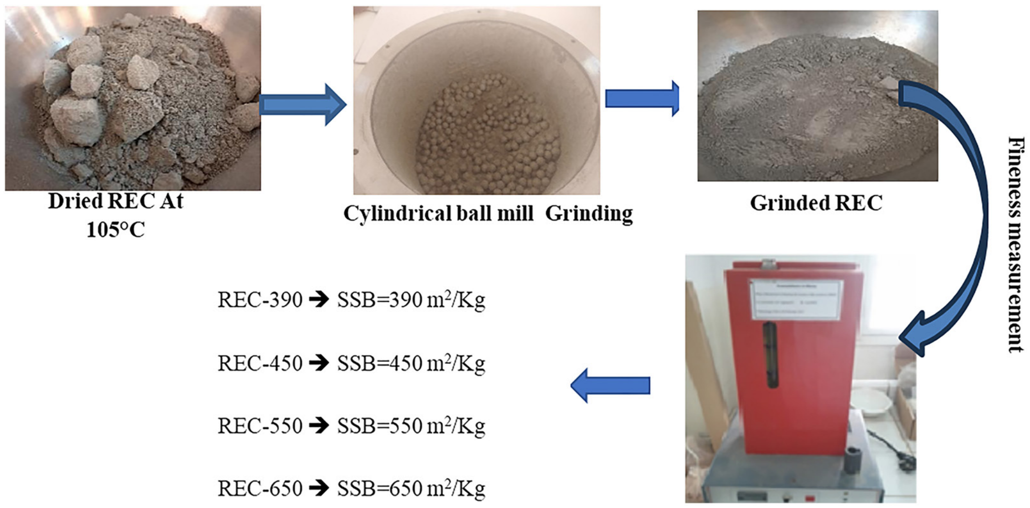

Figure 3: Grinding process for REC.

Table 2: Fineness of obtained REC against time and energy of grinding process.

SAMPLES	FINENESS (m²/kg)	TIME (MINUTES)	CONSUMED ENERGY (Wh)
REC 390	390	30	150
REC 450	450	60	300
REC 550	550	160	801
REC 650	**650**	360	1800

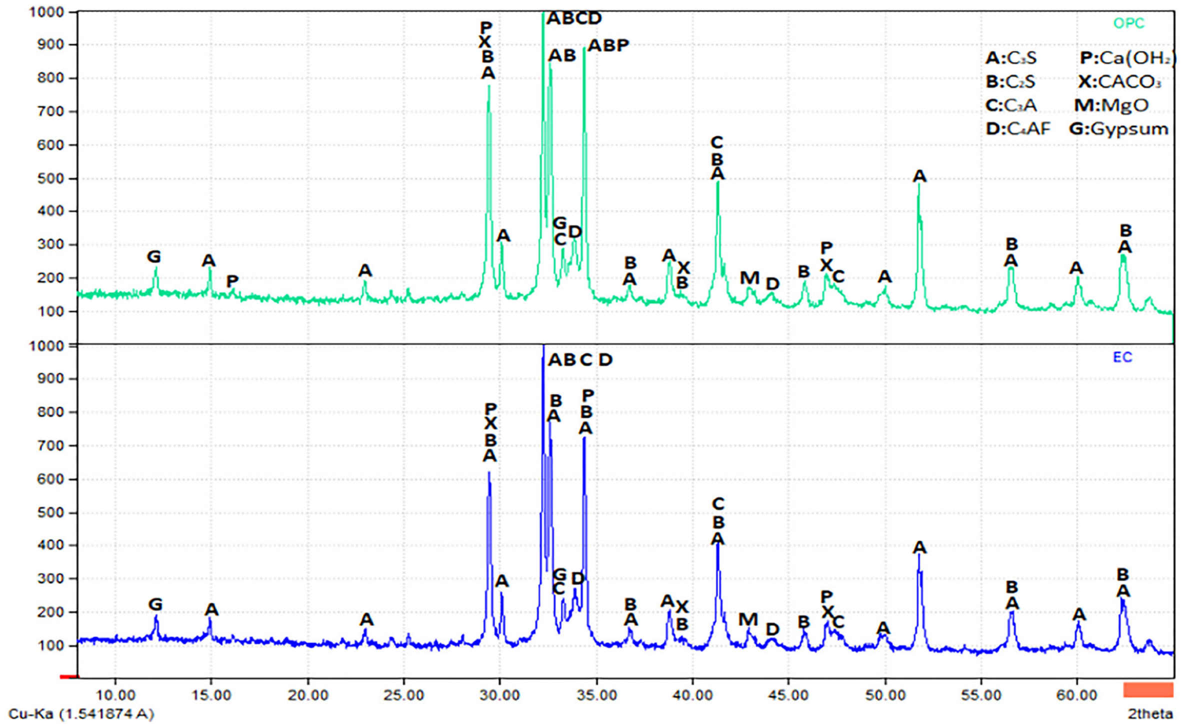

Figure 4: XRD analyses of OPC and EC.

analysis (DTA) under controlled atmospheric and temperature conditions. This apparatus is particularly appropriate for the examination of the thermal stability, decomposition reactions, and hydration behaviour of cement-based materials.

Figure 5 shows the Differential Thermal Analysis (DTA) and Thermogravimetric Analysis (TGA) of OPC and an expired sample. These thermal analyses provide relevant information on the type, stability,

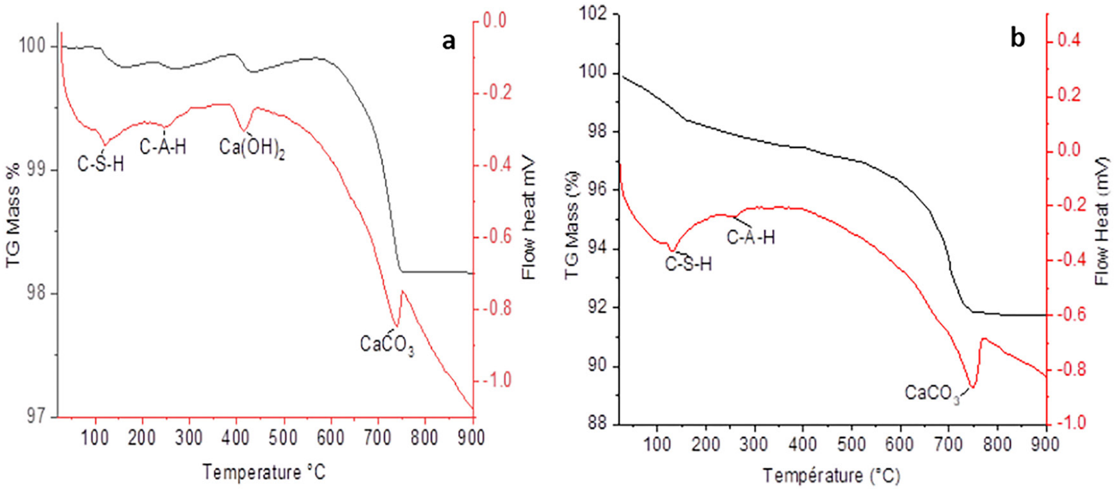

Figure 5: OPC (left) and REC (right) cements thermogravimetric analysis (TGA).

and level of hydration and carbonation reactions in samples; Typical thermal decomposition stages in cements can be deduced as follows:

- **C-S-H and ettringites dehydration (from 80–150 °C)**: The dehydration of calcium silicate hydrate (C-S-H) and ettringite is responsible for the mass loss observed in both OPC and REC samples at low temperatures. The OPC sample exhibits a diffuse endothermic peak and a mass loss of about 0.25%, which is indicative of the dehydration of ettringite and C-S-H. This mass loss indicates minimal prehydration and is consistent with the usual semi-crystalline structure of newly produced C-S-H [47]. The minor amount of pre-hydration products in this sample may appear during the manufacturing process, particularly when the clinker is ground. At this stage, the high temperatures encourage the dehydration of gypsum into more soluble calcium sulphates, such as bassanite or anhydrite, with the release of water [48]. Beside that the prehydrated cement (REC), on the other hand, shows a more significant mass loss in this area of about 1.85%, indicating the existence of more developed ettringite and C-S-H phases. The increase indicates there was partial hydration during storage, probably as a result of exposure to outside moisture, the cement's reactivity in future applications is reduced by this early hydration [49].
- **C-A-H Dehydration (200–300 °C)**: This temperature range is due to the decomposition of calcium aluminate hydrates (C-A-H), monosulfate (AFm) phases and hydrogarnet: C_3A hydration products [48]. In OPC, this event is observed as an endothermic peak with a corresponding mass loss of about 0.15%, according to normal development of hydration. In the expired cement, this peak is wider, relating to mass loss in the range of 0.5%, yet again supporting the presence of additional hydration products. The increased presence of aluminates in the expired sample confirms that C_3A has already reacted, reducing available reactive clinker phases for further hydration [49].
- **Portlandite Dehydroxylation (400–500 °C)**: One of the most typical differences between the two samples is observed in this region. For the new OPC, a distinct and strong endothermic peak and mass loss of approximately 0.15%, is due to the dehydroxylation of portlandite ($Ca(OH)_2$), one of the primary hydration products formed due to the reaction of C_2S and C_3S [50]. In the expired sample, this peak is significantly decreased or is nearly absent, indicating the $Ca(OH)_2$ consumption. This loss most likely results from pozzolanic activity and carbonation reactions under continuous exposure to CO_2 and humidity, leading to the formation of secondary products such $CaCO_3$ [51].
- **Decarbonation of Carbonates (700–800 °C)**: Under higher temperatures, decarbonation was also demonstrated for both specimens as indicated by an endothermic peak, due to decomposition of the calcium carbonate ($CaCO_3$) content. This peak is found in OPC, but it is relatively small, with mass loss of 1.7%, indicating that portlandite has experienced minor carbonation process during storage. However, with a mass loss about of 4.7%, the expired cement shows a noticeably sharper and more intense peak, indicating considerable carbonation. This shift shows that a large amount of $Ca(OH)_2$ has reacted already to form $CaCO_3$, reducing reactivity and affecting the structural use performance of cement negatively [52].

OPC cement contains more silicates (C_3S and C_2S), which offers greater early and long-term mechanical strength than REC cement as seen in Table 3. The Recycled cement with reduced silicate content could have sub-standard mechanical performance [53].

Table 3: Mineralogical composition.

MASSE PERCENTAGE %	C_3S	C_2S	C3A	C4AF	C-S-H /AFT/C-A-H	$Ca(OH)_2$	$CaCO_3$
OPC	62.18	7.52	6.47	8.29	0.55	0.12	3.68
REC	55.83	1.45	5.19	6.85	7.85	–	12.87

REC cement contains many more hydrated phases than OPC, indicating that it is already partially hydrated. This is due to the pre-hydration of the cement during storage [48]. Advanced carbonation of the recycled cement is indicated by the absence of portlandite and the high calcium carbonate content [54].

3.3. Formulation of standard mortar

Mortars were prepared according to the EN 196-1 standard [46]. Considering 450 ± 2 grams of cement, 1350 ± 5 gr of sand and 225 ± 1 gr of water, the water/cement ratio was kept constant at 0.5. Prismatic samples of size (40× 40 ×160) mm^3 were used to test the compressive strength according to EN 12390-1 [55] and EN 12390-2 [56].

All moulds were covered with plastic sheets and stored in the laboratory for 24 hours before the removal of the formwork. Subsequently, they were cured in water at 20 ± 2 °C. The tests were conducted at different ages of 3, 7, and 28 days. Additionally, to demonstrate strength development over time, all experiments were conducted on three specimen replicates.

3.4. Heat of hydration

Understanding the heat emitted during hydration is crucial for comprehending cementitious material's characteristics. In this study, the heat of hydration was measured using semi-adiabatic calorimetry. This method involves inserting a sample of freshly prepared mortar into a calorimeter, enabling the temperature evolution of the sample to be monitored and the amount of heat released to be determined as specified in the EN 196-9 standard [57]. The heat of hydration of the cement contained in the sample at a given moment is thus the sum of the heat accumulated in the calorimeter and that dissipated to the environment during the test. The thermal rise of the mortar is then compared with that of an inert sample placed in a reference calorimeter. The semi-adiabatic calorimetric method is particularly suitable for this purpose, as it facilitates simultaneous monitoring of the heat of hydration development of several mortar formulations under identical climatic conditions (20 ± 2°C). The installation is relatively simple, comprising an insulated calorimeter, a thermocouple (temperature sensor) and a data logger. The test specimen should possess a total mass of (1,575 ± 1) g. Each batch of mortar should be meticulously prepared with (360.0 ± 0.5 g of cement, (1,080 ± 1) g of sand in accordance with EN 196-1 [46], and (180.0 ± 0.5) g of distilled or deionised water. The test was conducted on five cement samples: OPC Cement, REC-390, REC-450, REC-550 and REC-650. The ensuing results are illustrated by the curves in Figure 6.

3.5. Isothermal parameters

The test of isothermal calorimetry, usually employed to analyse the kinetics of cement hydration, are performed under a constant temperature. These tests directly measure the rate of heat evolution for each sample by tracking the produced reaction heat flow. The total heat released over time can be calculated by integrating the recorded heat flow rate.

Cement setting is typically preceded by an early acceleration phase, which is identified by a peak in heat emission. The quick hydration of silicates causes this reaction. The hydration process then undergoes a deceleration phase, where it is regulated by diffusion instead of chemical control [48]. Therefore, it is essential to understand and interpret the heat of hydration curve following a calorimetry test in an accurate and efficient manner. In this study, calorimetry-based setting times have been established to facilitate the interpretation of test results based on the European standard EN196-3 [58]. These parameters also provide valuable indications for predicting concrete performance and ensuring quality control [59].

3.6. Compressive strength

In order to measure the compressive strength according to EN-196-1 [46], 40 × 40 × 160 mm^3 mortar specimens were prepared. Measurements were performed after 3, 7 and 28 days.

3.7. Evaluation of results using TOPSIS method

Both the fresh and hardened characteristics of the recycled cement mixtures were taken into account while evaluating the experiment's results. The objective was to determine which choice performed the best by

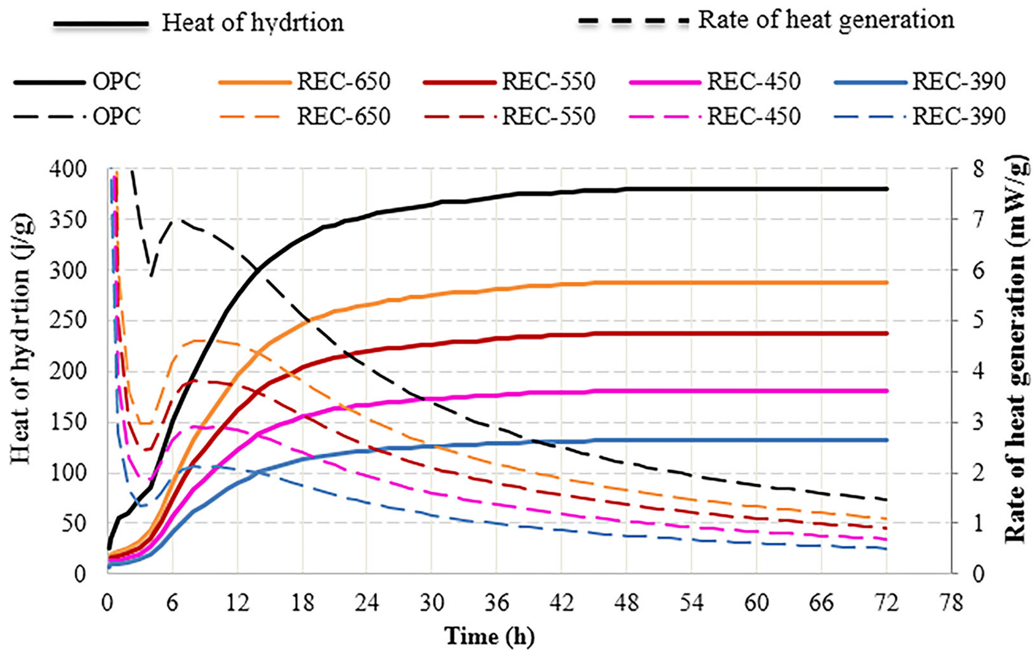

Figure 6: Semi-adiabatic calorimetry showing heat-flow and cumulative-heat curves.

examining all test data collectively in an accurate and impartial method. The TOPSIS approach, a well-known decision-making tool that assists in ranking possibilities based on a number of criteria, was employed to do this. It is frequently used in a variety of disciplines, including sustainability studies, engineering, and materials science [60].

The TOPSIS was used in this study to assess cementitious binders that were mechanically activated. Seven factors were taken into account in the analysis: energy consumption, compressive strength at 28 days, heat release rate, cumulative heat, initial and final setting times, and W/C ratio. Heat release, cumulative heat, and compressive strength were considered benefit criteria (higher values sought), whereas W/C ratio, setting times, and energy were considered cost criteria (lower values preferred).

First, the decision matrix was normalized using min–max normalization, which scaled each value from 0 to 1 according to its dataset minimum and maximum. A reverse scale was then used to modify the cost requirement. Every criterion was given the same weight of $w_j = 1/7$, which reflects an equitable evaluation.

Following the estimation of weighted normalized values, the ideal best (A^+) and ideal worst (A^-) solutions were identified. After calculating each alternative's distance from these ideal points, the TOPSIS relative closeness score (C_i) was determined using the conventional Equation 1:

$$C_i = \frac{D_i^-}{D_i^+ + D_i^-} \tag{1}$$

Where D_i^+ is the Euclidean distance to the ideal best, and D_i^- is the distance to the ideal worst, both parameters are given in Equation 2.

$$D_i^+ = \sqrt{\sum_i (\nu_{ij} - A_i^+)^2},\ D_i^- = \sqrt{\sum_i (\nu_{ij} - A_i^-)^2} \tag{2}$$

Based on C_i, the alternatives were rated; higher values denoted more ideal performance.

4. RESULTS AND DISCUSSION

4.1. Consistency test and setting time

The water demand (W/C) and setting time parameters for the OPC, EC, and mechanically reactivated cement samples are shown in Table 4. The result shows how cement age and mechanical reactivation have a major impact on workability and hydration kinetics.

Table 4: Consistencies and setting times results.

CEMENT	WATER DEMAND W /C	INITIAL SETTING TIME min	FINAL SETTING TIME min
OPC	0.290	210	265
EC	0.270	285	335
REC-390	0.280	276	333
REC-450	0.285	242	312
REC-550	0.290	231	308
REC-650	0.310	220	292

4.2. Water demand

OPC has a water demand of 0.29, which is within the typical range of marketed Portland cement [59]. While reactivated expired cement shows a higher values ranging from 0.28 to 0.31, this increase can be recognized to their Blaine finesses and chemical compositions. CANEDA-MARTÍNEZ *et al*. [61] revealed that the water demand increase with the incorporation of recycled materials as eco-cement, because of their porosity and high specific area leading to high water absorption rate. Moreover, specific surface area is the main parameter for assessing water requirements, but it is not sufficient to explain the variations observed.

It is also essential to consider particle morphology (shape and texture) and particle size distribution, the formation of hydrates on the surface of pre-hydrated cement particles results in increased roughness and an interlocking effect, thus increasing the effective surface area of cement particles. As a result, a greater quantity of water is required to obtain a paste of normal consistency [62]. This is particularly true of EC cement and probably REC-390 cement, whose insufficient grinding does not eliminate the roughness of the particles. REC-650 cement requires a higher quantity of water than REC-450 and REC-550, due to its greater specific surface area. SUN *et al*. [63] pointed that the cement paste hydrates more quickly and has less porosity because finer particles that have more surface area accessible for hydration, on the other hand, it can be assumed that the grinding of REC-450 and REC-550 cements favours a better particle size distribution, leading to a denser arrangement of the cement paste and, consequently, a reduced water demand.

4.3. Setting time

As seen in Table 4, the OPC has an initial setting time of 210 minutes, which aligns with the Portland cements standards [58,64]. In contrast, the REC setting time is ranging from 220 to 285 minutes. The shortest time is recorded for the mixture made incorporating REC-650; CANEDA-MARTÍNEZ *et al*. [61] reported that pastes with recycled cement presented longer initial setting times compared to those with ordinary Portland cement, attributed to the pre-hydration of recycled cement particles and their lower reactivity. LI *et al*. [65] found that the hydration process is more intense with finer particles, leading to faster setting times, especially at lower w/c ratios.

According to previous investigations, the setting time is extended in pre-hydrated REC cement due to the reduced reactivity of the pre-rehydrated cement. This reduction is attributed to the formation of stable phases such as C-S-H, C-A-H, and low-solubility gypsum in water, resulting from the pre-hydration of bassanite and/or anhydrite [10,12,66]. These hydration products form a very thin protective layer on the surface of cement grains, which hinders their normal hydration. Additionally, gypsum formation disrupts the sulfate balance, delaying ettringite formation and, consequently, the setting process [12].

On the other side; the final setting time of the control cement is achieved by 265 minutes, indicating a fast setting process in comparison with REC cements. Recycled cements present longer final setting times, ranging from 292 to 335 minutes. This phenomenon can be attributed to the slower hydration kinetics, which is typical for recycled materials with altered chemical composition or incomplete hydration. The shorter final setting time is recorded REC-650 with 292 minutes showing a good reactivity compared to other REC types, CRUZ *et al*. [67] observed that recycled cement pastes exhibited longer final setting times compared to Portland cement pastes, which was linked to the high water demand and pre-hydration of the recycled cement. Burris and Kurtis indicated that the setting time is also influenced by the composition of the cement, where higher sulfate content can shorten setting times, while increased w/c typically prolongs them [68].

GETACHEW *et al*. [69] revealed that the setting time delay is mainly due to the lower fineness and agglomeration of recycled particles. In this case, we have concluded that is complex interactions between water demand, recycled materials and cement properties.

4.4. Calorimetric evolution curves

By using semi-adiabatic calorimetry technique, the effects of grinding on the hydration of cement at an early age within 72 hours were examined; the heat of hydration and the rate of heat generation are illustrated in Figure 6. As shown, notable variations in the cumulative heat release behaviour of the different cementitious systems over a 72-hour period. Ordinary Portland cement with a high clinker percentage has a high degree of early-age reactivity, as demonstrated by OPC's steep initial slope and maximum cumulative heat of hydration, which reached about 370 J/g. With a comparable rapid heat evolution during the first 24 hours and a total heat release of roughly 275 J/g, REC-650 showed the most similar behaviour to OPC among the recycled cement blends. Increasing the specific Blaine surface, led to generate faster the heat of hydration, and enhances the intensity of chemical reactions. This suggests a comparably high proportion of reactive phases. While moderate cumulative heat release (240 J/g) was seen in REC-550, and despite a somewhat lower initial hydration rate than REC-650, it still showed significant responsiveness. Scrivener *et al*. concluded that by producing finer particles, mechanical grinding improves the cement-water interaction and accelerates hydration reactions and reported that the dormant period length depends on particle size and reactivity [46], GHOLIZADEH-VAYGHAN *et al*. [70] assert that early hydration heat helps cements to develops strength rapidly.

Besides that, both REC-450 and REC-390 showed noticeably slower initial hydration kinetics and much lower total heat release values, at about 190 J/g and 160 J/g, respectively. These patterns imply that the availability of reactive clinker phases is gradually decreasing or that less reactive components. Its poor hydration potential is further shown by the fact that REC-390's heat release plateaued earlier than that of other blends. Also this can be attributed to the lower specific Blaine surface limiting the particle-water interactions; coarser cement particles hydrate more slowly leading to prolonged induction periods and reduced peak heat release [53].

4.5. Compressive strength

The cement's specific Blaine surface play a critical role in optimizing the material performance of cement based materials for construction applications. Figure 7 shows how the compressive strength of OPC changes throughout a 56-day curing time in comparison to reactivated cement systems (REC-650, REC-550, REC-450, and REC-390). High clinker content and quick hydration kinetics were the primary characteristics of OPC, which continuously demonstrated the highest strength values, reaching roughly 45 MPa at 56 days and a rapid increase during the early curing period (0–7 days). The most comparable performance was shown by REC-550 and REC-650 mixtures, where they reached approximately 35 and 36 MPa at 56 days and exhibited a strength gain pattern that closely matched that of the reference cement, especially in the early age (first 7 days), providing good reactivity and favourable microstructural development. According to anterior studies, mechanically activated cements have better hydration kinetics and particle interaction, which results in stronger compressive strengths [71–73]. Cement grinding can be optimised to form an effective arrangement, thus facilitating the successful mitigation of the disadvantages associated with the relatively low hydraulic properties of expired cement, it is evident that this process has enhanced the CSH and diminished the quantity of water that remains following hydration, and reduce the water-filled size and enhance the strength of the material [74].

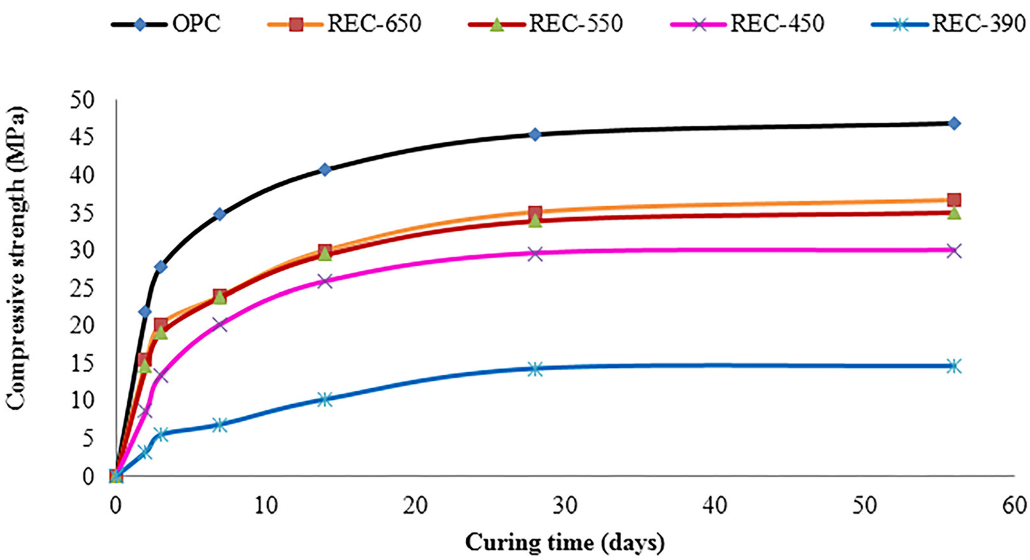

Figure 7: Compressive strength curves of cement samples.

The compressive strength of REC-390 was the lowest over the testing period, reaching a plateau of about 18 MPa on day 56. Its restricted hydration potential, as seen in the calorimetry data (Figure 7), is consistent with this noticeably poorer performance, which most likely indicates a high degree of clinker replacement with low-reactivity components.

Prehydrated cement loses compressive strength, particularly when exposed to increased humidity levels, which can prevent strong bonding from forming during the hydration process. Additionally, prehydration causes minimal strength losses during future hydration at 60% relative humidity, whereas early-age compressive strength significantly decreases at 85% relative humidity [12].

The Figure 8 shows a strong relationship between the reactivated cement's compressive strength and heat of hydration at 24 and 72 hours (R2 = 0.9824 and 0.9619, respectively). Rapid early strength development at 24 hours is correlated with a higher heat of hydration, indicating strong exothermic responses.

By 72 hours, the heat release has decreased, suggesting that as cement ages, hydration is slowing down. This pattern indicates that reactivated cement provides quick early strength gain while preserving long-term durability, with performance optimization largely dependent on the initial reactivity. This analysis illustrates the utility of semi-adiabatic calorimetry method for tracking hydration kinetics and predicting early compressive strength, which is essential for optimising the performance of cementitious materials [75].

4.6. TOPSIS optimisation result

As seen in Table 5, the REC 550 mixture was characterized as the most ideal among the recycled cements evaluated under the standard TOPSIS analysis with equal weighting. This mix was able to achieve a balance between mechanical strength (35 MPa), heat evolution (240 J/g), and energy demand (801 Wh), with setting times that

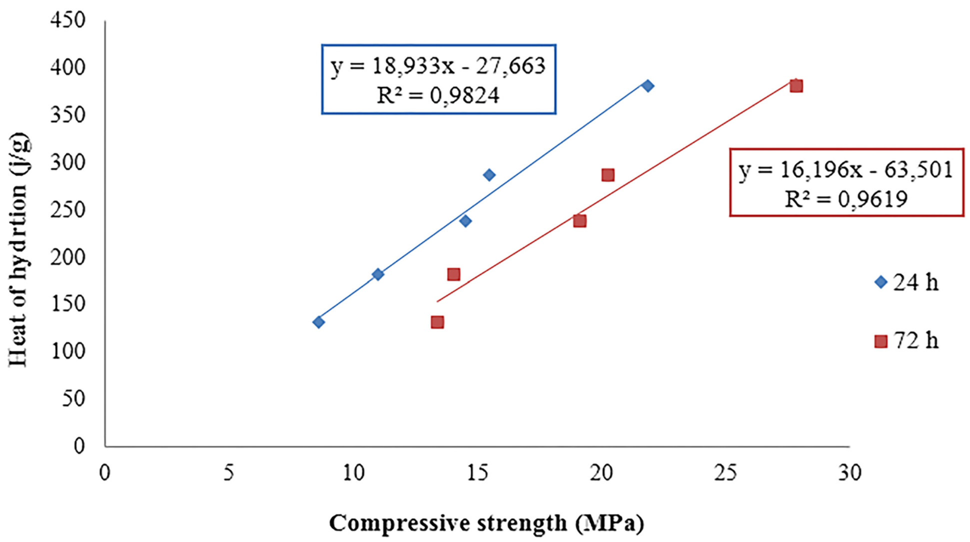

Figure 8: Thermal- compressive strength correlation of cement samples.

Table 5: Final standard TOPSIS results.

CEMENT	W/C	INIT SET	FINAL SET	HEAT RATE PEAK (mW/g)	CUMULATIVE HEAT (J/g)	28 DAYS STRENGTH (MPa)	ENERGY (Wh)	TOPSIS WEIGHT SCORE	RANK
REC 390	0.28	276	333	2.1	135	15	150	0.387	4
REC 450	0.285	242	312	2.9	180	30	300	0.580	3
REC 550	**0.29**	**231**	**308**	**3.9**	**240**	**35**	**801**	**0.703**	**1**
REC 650	0.31	220	292	4.7	290	36	1800	0.613	2

are comparatively close to those for OPC. While REC 650 exhibited slightly higher strength, it consumed much more energy during its functional period, which diminished its total desirability. These findings highlight REC 550 as an efficient recycling circuit capable of providing competitive performance characteristics similar to those of OPC, without compromising technical quality.

In this study, we carried out an evaluation of the energy consumption required to reactivate expired Portland cement. Our results show that the reactivation process consumes approximately 8 kWh per ton of recycled cement. When compared to the total energy required to produce conventional cement estimated between 800 and 1200 kWh per ton [76] this represents a very small fraction, approximately 0.8%. Notably, around 50 kWh/t is typically required for final clinker grinding alone, further highlighting the energy efficiency of our proposed approach.

The objective of our work is to promote the use of expired (prehydrated) cement in the public works and construction sectors. The reactivation method we propose is simple, cost-effective, and enables the recycled cement to meet the 32.5 strength class requirement as defined by the Algerian standard NA 442 (refer to Table 1) [64]. This class of cement can be effectively applied in cement plaster coatings, reinforced concrete platforms, and slab paving, among other uses. We believe that our solution offers a practical and scalable approach for both the cement industry and broader waste recovery efforts, contributing to sustainability and circular economy initiatives.

5. CONCLUSIONS

This study investigates the influence of grinding on the physical and mechanical properties of expired cements in order to determine their reactivation potential. The experimental results allow for the formulation of the following conclusions:

- The hydration kinetics and strength performance of expired cement are enhanced by mechanical grinding, particularly as fineness increases.
- Compared to fresh OPC, recycled cements show delayed setting periods; however, this difference is diminished by finer grinding.
- By using thermal response analysis, semi-adiabatic calorimetry demonstrated to be a useful and accurate technique for predicting early-age compressive strength.
- X-ray diffraction (XRD) and thermal (TGA/DTA) tests verified the development of hydration products in the EC as well as the partial consumption of the primary clinker phases that were initially present in OPC.
- REC has a lower heat of hydration than OPC, making it potentially suitable for massive structures such as dams and deep foundations.
- The grinding process present a considerable enhancement of the physical and mechanical properties of expired cement samples
- The most effective blend was found to be REC 550, which offered a good balance between strength (35 MPa), consumption of energy (801 Wh), and hydration behaviour.
- The potential of REC to promote the circular economy and contribute to a reduction in carbon footprint is significant.

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