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Utilization of waste foam concrete with MPCM as a substitution material for cement in mortars

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ABSTRACT

Given the rising popularity of foam concrete (FC) for both structural and insulating purposes, evaluating the feasibility of recycling after its lifespan is crucial in the context of the growing emphasis on sustainable building practices. One approach to recycling FC incorporating microencapsulated phase change material (MPCM) involves utilizing recycled foam concrete powder (RFCP) as an additive in cement composites. This article aims to investigate the impact of RFCP without and with MPCM when employed as a partial replacement for cement in mortars. Furthermore, the study verifies various processing methods such as crushing, grinding, and heat treatment for RFCP. The results reveal that introducing RFCP, regardless of the MPCM presence and processing method, significantly affects the properties of both cement and mortar. The presence of MPCM in RFCP negatively influences the flowability of fresh mortars, delays the setting time, and reduces the hydration heat within the first 48 h. However, the presence of MPCM does not significantly affect mortars' strength and water absorption but simultaneously it increases shrinkage and decreases thermal conductivity. Grinding RFCP mitigates the adverse effects of MPCM, while thermal processing removes MPCM from RFCP, albeit with an associated increase in water demand. A noteworthy finding is that mortars having 20 % RFCP, with or without MPCM, exhibit compressive strengths exceeding 16 MPa and 42.5 MPa after 2 and 28 days, respectively. These results meet the requirements outlined in EN-196-1 for cement of class 42.5, highlighting the potential to produce CEM II/A-F 42.5 using RFCP with MPCM.

1. Introduction

An important aspect in the development of sustainable building practices lies in addressing the challenge of managing demolition waste at the end of a material's lifecycle. This topic aligns with the ongoing standardisation efforts and regulations in the European Union (EU), aiming to the implementation of Environmental Product Declarations (EPDs). In 2022, the EPD standard underwent a revision to EN 15804 + A2 [1], now requiring the inclusion of end-of-life impact calculations and extending the scope to encompass benefits beyond the system boundary. This expanded scope includes considerations such as the benefits derived from reuse, recovery,

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and recycling processes [2]. Although the publication of EPDs is currently voluntary, it should be noted that some manufacturers of construction materials are already proactively releasing EPDs for their products as a means of showcasing the sustainability efficacy of their offerings. Simultaneously, it is anticipated that the EU will present the updated Construction Products Regulation later this year, mandating the inclusion of EPDs in the future [3]. Thus, each material used in construction will have to undergo assessment for its recyclability. This necessity naturally extends to cement-based materials. Cement-based materials, including concrete, cement mortars, and increasingly popular foam concrete, rank among the most often used construction materials globally. Recycling these materials not only diverts waste from landfills but also conserves natural resources by diminishing the demand for new raw materials. Therefore, the reuse of cementitious materials from demolished elements and structures contributes to a more sustainable construction industry, building up a circular economy within the construction sector.

In general, recycling cement-based materials can be approached in two ways, depending on their specific characteristics and properties. For instance, in the case of conventional concrete, there are two primary methods of reuse: either incorporating whole elements in their original state for constructing new buildings or after crushing, integrating them into concrete as coarse or fine aggregates, or as recycled concrete powder (RCP) [4]. While similar recycling methods can be applied to foam concrete (FC), they do present certain challenges. Reusing whole elements becomes complicated, if not entirely impossible, due to the typically low strength of FC, especially in ultralight foam concrete (ULFC).

Generally, the potential applications of recycled concrete powder (RCP), made of conventional concrete (CC) or foam concrete (FC), are particularly promising and encompass various directions. Firstly, RCP can serve as an ingredient in cement, with the possibility of incorporating up to 40 %, following the standard EN 197-6 Cement - Part 6: Cement with recycled building materials [4,5]. Secondly, RCP finds utility as an ingredient in mortars, concretes, and foam concrete [6], where it can be introduced at levels ranging from 20 % to 40 %, effectively replacing a portion of the cement and/or aggregate powder fractions. Additionally, RCP can be employed as (1) a raw material component for producing cement clinker [7–9]; (2) as part of lightweight artificial aggregates through granulation [10,11]; (3) as a foundational element for producing special binders [12,13]; (4) as a component of geopolymers [5,14].

RCP can be utilized either in its original form directly from the crushing of CC/FC or after sieving to attain a finer particle-size powder. However, this RCP typically exhibits high water absorbability due to the pronounced porosity and irregular shape of its grains [5]. Enhancements in RCP properties concerning water absorbability can be done through processing [5]. Two fundamental processing methods for RCP exist: mechanical processing involving grinding and thermal treatment [5]. In the grinding method, RCP is ground to a specific degree, chosen to function as a micro filler without being excessively fine, as overly fine particles may result in material agglomeration [5,15,16]. The grinding process initially targets the large, porous grains of RCP, thereby reducing its water absorbability [5,15,17,18]. With increasing grinding time, the irregular, crushed-aggregate-like grains of RCP gradually transform into a more spherical shape [5,15,16]. Additionally, grinding may activate non-hydrogenous cement parts and certain hydrates (particularly those containing SiO₂) also may adopt an amorphous form, increasing the activity of RCP [5,15,17,18]. Grinding processing is conveniently applicable in industrial cement production. In the heat treatment method, RCP undergoes exposure to elevated temperatures, leading to the dehydration of cement. Research findings from Refs. [5,12,19–21] indicate that the optimal temperature range for heat treating RCP falls between 600 and 800°C. Within this temperature range, Ca(OH)₂ undergoes almost complete decomposition, while hydrated calcium silicates (C–S–H) and CaCO₃ experience significant decomposition. The resultant active phases and oxides, with a high specific surface area, enhance the activity of RCP. Thus, when incorporated into cement composites, these treated RCP particles can contribute to the improvement of composite properties [5,12,19–21]. It is important to acknowledge that while heat treatment offers these benefits, it also presents technical and economic challenges and has a negative environmental impact. Other methods of RCP processing include: (1) blending RCP with other materials such as ground brick waste, fly ash, lime, and ground limestone [5,10,15,17,18,22–27], and (2) subjecting RCP to carbonation through curing in carbon dioxide [5,28,28–32]. While these approaches offer the potential to mitigate the adverse effects of RCP on the properties of both fresh and hardened new composites, they currently face challenges in terms of technology and economics, hindering their broader practical implementation [5].

The effect of RCP obtained from conventional concrete on the properties of mortars and concretes has been extensively studied. It is noteworthy that in most of these studies, RCP was incorporated either as a substitute for a portion of the cement or as a component of the cement itself. Consequently, these studies have led to the inclusion of the possibility of using RCP as a main constituent of cement in the EN 196-1 standard [33,34], indicating a relatively well-established recognition of RCP usage. Analysing the impact of RCP on cement-based materials, the presence of RCP in quantities below 10 % of the cement typically has minimal impact on the properties of both fresh and hardened composites [5,35]. If RCP is used in larger amounts, then its effect is usually negative. The incorporation of 10 %–45 % RCP as a substitute for cement worsens the flowability in fresh mortars and concretes, with the degree of worsening correlating with the RCP content [5,22,36–45]. Studies [32,38] revealed that adding 20 %–40 % RCP leads to an increase in the yield stress and plastic viscosity of mortars, with a more rapid increase in these parameters over time. It is noteworthy that while the impact of RCP on flowability is generally disadvantageous in conventional applications, for some self-compacting concrete (SCC) mixes and cement composites used in 3D printing, the incorporation of RCP may be considered desirable. Due to its high-water absorption ability, RCP can serve as an effective stabiliser, offering a cost-effective alternative to more expensive mineral additives in this role. Positive effects of RCP on the stability of SCC mixes were documented in Refs. [39,46]. The possible negative impact of RCP on the flowability of cement composites can be mitigated by subjecting it to grinding processes [5,17,18,40]. On the other hand, heat treatment significantly enhances the water-absorbing properties of RCP, thereby intensifying its adverse effect on the flowability of fresh composites [5, 12,19–21,46,47]. Due to its limited chemical reactivity, substituting part of the cement with RCP delays also the setting time and reduces the amount of heat released during the process [42]. Further, the inclusion of RCP in composites leads to a reduction in density and an increase in porosity, as evidenced by various studies [28,40,42,48,49]. For instance, research indicated that the porosity of a

mortar containing 20 %, 40 %, and 60 % RCP after 28 days increased by 6.3 %, 16.5 %, and 26.6 %, respectively [40]. This rise in porosity adversely affects the strength and durability of the composites. Up to a 20 % addition of RCP, the decrease in compressive strength for mortars and concretes does not typically exceed 10–15 %. However, higher amounts result in a swift rapid proportional decline in strength [15,16,18,22,28,36,37,39–43,45,48–51], especially beyond the 30 % RCP. RCP exerts a comparable influence on flexural strength [22,36,37] and elastic modulus [22,36]. Further, the introduction of RCP delays strength development during the initial hardening period [22,36,37,40,45]. Concrete shrinkage increases with higher RCP content, reaching approximately 30 % higher value than that of reference concrete at 30 % RCP addition [39]. The presence of RCP negatively affects concrete durability, significantly increasing carbonation depth [22,39], decreasing frost resistance [22], and typically elevating water absorption [18,37,40], though grinding-processed RCP may exhibit lower water absorption and capillary suction due to its filler effect [22]. RCP addition also lowers the resistivity of cementitious composites [40,49]. Improved effects of RCP addition to mortars and concretes can be achieved when using grinding-processed or heat-treated RCP. Composites with processed RCP exhibit lower porosity and a tighter structure than reference composites, as long as the RCP addition stays below 30 % [15,17–20,50,52]. Furthermore, mortars and concretes with heat-treated RCP at 600–800 °C exhibit higher strength than those processed at lower or higher temperatures, as reported in studies [19,20,52]. However, a higher addition of heat-treated RCP can result in properties inferior to those of the reference material [50]. When analysing the adverse effects of RCP on hardened composites, it is crucial to recognize its often-associated negative impact on mixture flowability and the method of modifying the mixture composition to achieve adequate workability. Considering the effective water-cement (w/b) ratio in the design and compensating for the loss of flowability caused by RCP with high-range water reducer (HRWR) rather than increased water content helps mitigate the impact of RCP on hardened composite properties, rendering it more controllable.

Simultaneously, when examining the effects of adding RCP, it is crucial to consider that the properties of RCP depend on various factors, including the age, composition, and properties of the element from which it was derived, the conditions under which it was used, and the degree and progression of its physical and chemical degradation processes, particularly the presence of harmful substances like chlorides and sulphates. Consequently, RCP may exhibit considerable variability not only across different sources but also within a single source. Moreover, in the case of RCP obtained from ultralight foam concrete (ULFC), which primarily consists of a cement paste with a hydrated binder and usually a trace amount of unhydrated binder [53], the characteristics of RCP can differ quantitatively from that of RCP sourced from conventional mortar or concrete. Therefore, the introduction of RCP from ULFC into new cement composites may yield quantitatively distinct effects compared to RCP from ordinary mortar or concrete. Furthermore, recycled concrete powder (RCP) derived from FC or ULFC elements can include phase change material (PCM), as successful attempts are underway to develop ULFC with PCM and densities ranging from 200 to 400 kg/m³ [54–60], intended for use as thermal insulation material [61,62]. This inclusion of phase change material is typically implemented using microcapsules, which have diameters ranging from 1 µm to 1 mm. The microcapsules consist of a phase change material (PCM) as the core, typically enclosed within a polymeric capsule [63–65]. In building construction, paraffin-based organic PCM is commonly used as the core material within microencapsulated phase change material (MPCM) [63–67]. Furthermore, if MPCM is present in the ULFC, it will also be present in the RCP, inevitably affecting its properties. It can be also anticipated that during the crushing process, PCM microcapsules may be damaged, releasing the PCM (typically paraffin) in its free state, although conclusive data in this regard is currently lacking. Notably, during the heat treatment of RCP, paraffin-based MPCM can be extracted as they burn at a relatively low temperature of about 400°C. In this case, this heat treatment process enables the RCP to be paraffin-free, thereby avoiding potential adverse effects on its properties.

Therefore, such recycled foam concrete powder (RFCP) possesses distinct properties attributed to its composition and the possible inclusion of MPCM. Considering the additional influence of MPCM present in RFCP and drawing insights from the summarized experiences of using conventional RCP, it becomes evident that incorporating RFCP derived from ULFC will adversely impact both the fresh and hardened properties of new composites. When assessing the effects of MPCM, it becomes crucial to ascertain the amount of MPCM introduced into the new composite along with RFCP. If recycled concrete powder is sourced from ULFC with a density of 200–400 kg/m³ containing 20 % MPCM by the volume of the base paste (approximately 4 % by the volume of foam concrete), the MPCM content in the RFCP will be approximately 10 % by weight of RFCP. This quantity seems to be insufficient to significantly impact the properties of cementitious composites. However, it is essential to note that these suggestions primarily pertain to cases where the microencapsulated PCMs are mostly undamaged. In the case of RFCP with MPCM, it is reasonable to anticipate that a significant portion, if not all, of the MPCM (paraffin) will be released from the capsules due to the mechanical methods employed during its extraction. This might result in a more pronounced negative impact of MPCM than expected from its relatively small quantity.

It is important to highlight the impact of recycled foam concrete powder (RFCP) containing MPCM on the properties of cementitious composites has not been systematically studied, and there is a lack of work describing the comprehensive impact of replacing cement with recycled foam concrete on a diverse range of mortar properties. Moreover, there is a notable absence of investigation into the potential and effects of processing RFCPs with MPCM using methods such as grinding or heat treatment, which facilitate the removal of MPCM from RFCPs. Given the rising popularity of foam concrete (FC) for both structural and insulating purposes, evaluating the feasibility of recycling after its lifespan is crucial in the context of the growing emphasis on sustainable building practices.

This necessity, coupled with the existing knowledge gap, which notably lacks research on foam concrete waste with MPCM, prompted the undertaking of research with the objectives of (1) determining the impact of recycled foam concrete powder (RFCP) derived from ultralight foam concrete (ULFC), without and with the addition of MPCM, on mortar properties, and (2) analysing the possibilities and effects of processing the mentioned RFCP, with and without MPCM, through grinding or heat treatment methods. It is important to emphasize that the use of RFCP in cementitious composites primarily aims at environmental benefits. It should be regarded as an additive to cement or concrete for recycling purposes rather than material valorisation.

2. Experimental program

2.1. General information

The impact of recycled foam concrete powder (RFCP), both with and without microencapsulated phase change material (MPCM), underwent comprehensive testing using standard mortars under EN 196-1 [33]. Thus, the results obtained can be interpreted concerning the utilization of RFCP as a component of cement following EN 196-6 [68] and/or as a Type I mineral additive introduced to replace a portion of the cement as per EN 206 [69]. The influence of RFCP without MPCM on mortar properties was juxtaposed with mortars made from ordinary Portland cement (CEM I) and those composed of 80 % CEM I and 20 % ground limestone LL (equivalent to CEM II/A-LL cement). Further, the effect of RFCP with MPCM on mortar properties was compared to mortars with RFCP lacking MPCM.

The tested recycled foam concrete powders (RFCPs) were derived from recycled ULFC panels with a density ranging from 200 to 350 kg/m³. The used ultra-light foam concrete was developed and tested under the European Commission-funded project NRG-STORAGE [61,62,70]. The production of ULFCs involved the pre-foaming method, where the base paste and foam were prepared separately and then mixed. The volume of the base paste in ULFC ranged from 13 % to 20 % [61,70–72,72]. The base paste, with a water-to-binder ratio of 0.40, comprised cement (CEM I 42.5 R or CEM I 52.5 R), metakaolin (~16 % of cement weight), MPCM up to 20 % of the base paste volume, and admixtures such as superplasticizer, stabilizing, and accelerating agents [61,71,72]. Two types of paraffin-based MPCM with a polymeric shell, namely Nextek 24D (PCM1) or Nextek 37D (PCM2), were utilized in ULFCs. The foam, with a density of 70 kg/m³, was generated using a protein foaming agent. Details regarding the properties of the ULFCs from which the RFCPs were derived can be found in Refs. [70–72].

2.2. Research plan

In the research, the influence of the following factors was investigated.

- Presence and quantity of RFCP in the mortar. RFCP was employed as a substitute for a portion of the CEM I cement (OPC) in two specified quantities: (1) 20 % by weight and (2) 40 % by weight. The selection of 20 % aimed to align with the maximum proportion of recycled concrete fines (F) in CEM II/A-F cement, while the use of 40 % was intended to assess the impact of incorporating RFCP over the maximum F content in CEM II/B-F cement (which is 35 %).
- Presence and type of MPCMs in the RFCP. The RFCPs utilized were derived from two sources: ULFC without the inclusion of MPCM, and ULFC with the addition of MPCM, specifically Nextek 24D (later designated as PCM1) or Nextek 37D (later designated as PCM2).
- Method of RFCP processing. Three techniques were employed to produce RFCP: (1) crushing and sieving of ULFC to a particle size <0.125 mm; (2) crushing ULFC followed by grinding and sieving to a particle size <0.063 mm; and (3) subjecting RFCP obtained through method (1) to heat treatment at 750°C.

Method (1) involved crushing naturally dried ULFC in a crusher and subsequently sieving the resulting material to achieve a grain size <0.125 mm. Following sieving, approximately 40 % of the RFCP with a grain size <0.125 mm and around 60 % of the waste with a grain size of 0.125–1 mm were obtained. This procedure allowed obtaining RFCP of specific surface area (SSA) around 6000 cm²/g.

Method (2) included crushing naturally dried ULFC in a crusher, grinding the obtained waste dust for 1 h in a Los Angeles mill, and then sieving the resulting material to reach a grain size <0.063 mm. After sieving, approximately 10 % of the RFCP with a grain size >0.063 mm remains, but it can undergo further grinding. Consequently, this method allows for the complete utilization of the waste. This method resulted in the RFCP of SSA 6000–7500 cm²/g.

Method (3) involves the heat treatment of RFCP obtained through method (1), specifically RFCP with a grain size <0.125 mm. This method was selected not only for the potential activation of RFCP but primarily as a means of removing MPCM from it. Since the evaporation temperature of paraffin is around 400 °C, subjecting RFCP with MPCM to a significantly higher temperature facilitates its removal. Drawing on prior experience with heat treatment of paste and RCP outlined in the Introduction, 750 °C was determined as the maximum heat treatment temperature. The heat treatment cycle comprises the following phases: temperature rise from 20 to 750 °C

Table 1
Research plan and sample notations.

Mortars with RFCP – with 20%wt or 40%wt RFCP content as cement replacement			
RFCP type	Method of RFCP processing		
	Crushing and sieving to a grain size <0.125 mm SSA ~6000 cm ² /g (RFCP_C)	Crushing, grinding and sieving to a grain size <0.063 mm SSA 6000–7500 cm ² /g (RFCP_G)	Heat treatment of RFCP_C at 750°C SSA >7500 cm ² /g (RFCP_T)
RFCP without MPCM	RFCP_C_0	RFCP_G_0	RFCP_T_0
RFCP with MPCM	RFCP_C_PCM1	RFCP_G_PCM1	RFCP_T_PCM1
Nextek 24D (PCM1)			
RFCP with MPCM	RFCP_C_PCM2	RFCP_G_PCM2	RFCP_T_PCM2
Nextek 37D (PCM2)			
Reference mortars without RFCP			
OPC – mortar with 100 % CEM I			
OPC + LL – mortar with 80%wt CEM I and 20%wt LL as cement replacement			

over 3 h, temperature maintenance at 750 °C for 3 h, and natural cooling for 24 h. While this method allows for the complete utilization of the waste, it carries a more substantial environmental impact compared to methods (1) and (2). As a result of heat treatment at 750 °C, RFCP from ULFC without MPCM experiences a weight loss of approximately 17 % due to dehydration of the paste components, consistent with existing literature data. Under the same conditions, RFCP with MPCM, specifically Nextek 24D and Nextek 37D, lose 27.8 % and 26.6 %, respectively. These levels indicate the presence of approximately 10 % MPCM in RFCP by weight, affirming the estimates discussed in the Introduction. It should be noted that the SSA of RFCP_T was the highest of all of the RFCP types, over 7500 cm²/g and reaching 9000 cm²/g.

The full research plan and sample notation are summarized in Table 1. It is important to highlight those investigations into the impact of incorporating 40 % RFCP were primarily conducted for cements and mortars with RFCP processed by grinding (RFCP_G). The introduction of 40 % RFCP_C or RFCP_T significantly diminishes the workability of the mortars, preventing the proper preparation of samples for testing the properties of hardened mortars.

2.3. Materials

Portland cement CEM I 52.5 R (OPC), limestone powder (LL), and RFCP with properties outlined in Table 2 and Table 3, along with CEN Standard Sand following EN 196-1 [33] for assessing the grain size distribution of cement, were employed. It is noteworthy that the fineness and specific surface area of limestone powder surpass those of cement but are notably lower than those of RFCP.

Additionally, it can be noticed that outside of cement, both limestone and RFCP have a high loss on ignition (LOI). In the case of limestone, this can be attributed to the calcination of calcium carbonate, which amounts to a mass loss of 41.2 %. In the case of RFCP, the observed high LOI relates to the water evaporation and phase decomposition occurring in high temperatures. As RFCP is produced from foam concrete, it was expected that in high temperatures dehydration of alumina phases may occur, as well as decomposition of portlandite and CSH phase [73–75].

The laser diffraction analysis (LDA) was used to showcase the particle size distribution of the RFCPs, OPC and LL in Fig. 1. Additionally, Fig. 2 displays scanning electron microscope (SEM) images of RFCP with two distinct grain sizes, namely <0.125 mm (SSA 5917 cm²/g) and <0.063 mm (SSA 6636 cm²/g), both incorporating MPCM Nextek 24D (designated as RFCP_C_PCM1 and RFCP_G_PCM1, respectively).

The laser diffraction analysis (LDA) reveals that the fineness of RFCP_C is notably lower compared to that of cement. Grinding significantly enhances the fineness of RFCP_G, although it remains lower than that of cement. Heat treatment increases the fineness of RFCP_T, albeit to a lesser extent than achieved through grinding. However, it is essential to note that LDA does not consider the porosity of RFCP grains and their irregular shape. SEM images (Fig. 2) illustrate the irregular shape, porous nature, and rough surface of the grains, highlighting the higher fineness of RFCP_G_PCM1 compared to RFCP_C_PCM1. As indicated in Table 2, RFCP_C exhibits a specific surface area approximately 30 % higher than cement, and grinding elevates the specific surface area of RFCP_G to approximately 45 % above that of cement. Heat treatment results in RFCP_T having a specific surface area as much as 70 % higher than cement. The MPCM content of Nextek 24D and Nextek 37D in RFCP_C and RFCP_G is approximately 10 % by weight. Furthermore, SEM analysis reveals that there are no free and intact MPCM microcapsules in both types of RFCP; only remnants of the microcapsules are observed (Fig. 2b and d). This is expected due to the significant forces used during RFCP production, which can damage or destroy the microcapsules, allowing paraffin from the PCM to coat the RFCP grains. Notably, in the case of RFCP_C_PCM1, small clusters of grains were found on the remnants of the microcapsules, as shown in Fig. 2b. Conversely, no similar elements were found in RFCP_G_PCM1 outside the broken capsules. This discrepancy may be attributed to the fact that through grinding processing, clusters of free paraffin are dispersed on the surface of the RFCP grains.

In Fig. 3, the thermal analysis results of RFCP are presented without the inclusion of MPCM in its composition (RFCP_C_0) and with a 10 % addition of MPCM Nextek 24D (RFCP_C_PCM1). Several prominent peaks are evident. Peaks in the range of 100–200 °C are associated with water evaporation, signifying the dehydration of C₃A and ettringite phases. An additional peak at approximately 350 °C results from the decomposition of alumina phases, while the peak at around 450 °C is attributed to the dehydroxylation of portlandite. The peak observed at 700–750 °C indicates the decarbonation or decomposition of carbonates present in RFCP. A smaller peak, noticeable at 617 °C in RFCP_C_0, can be linked to the presence of metakaolin in the foam concrete. This peak may be less apparent in RFCP_C_PCM1 due to the MPCM's presence, which reduces the volume of metakaolin and consequently makes the already

Table 2
Basic properties of OPC, LL and RFCPs.

Material	Specific surface, cm ² /g	Density, g/cm ³
CEM I 52.5 R (OPC)	4595	3.10
Limestone (LL)	4785	2.93
RFCP_C_0	5926	2.28
RFCP_C_PCM1	5917	2.09
RFCP_C_PCM2	6014	2.08
RFCP_G_0	7236	2.07
RFCP_G_PCM1	6636	1.96
RFCP_G_PCM2	6204	1.84
RFCP_T_0	7871	2.88
RFCP_T_PCM1	7636	3.24
RFCP_T_PCM2	9008	2.91

Table 3
Chemical composition of OPC, LL and RFCPs.

Binder	Chemical composition, % wt							
	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O + K ₂ O	LoI
CEM I 52.5 R (OPC)	20.10	4.50	3.30	64.90	1.40	2.8	0.93	0.66
Limestone (LL)	3.6	0.5	0.4	53.55	0.70	0.02	–	41.2
RFCP_C_0	16.2	3.8	2.8	55.2	1.2	2.24	0.70	17.1
RFCP_C_PCM1	14.2	3.4	2.5	48.34	1.0	1.9	0.61	27.8
RFCP_C_PCM2	14.7	3.5	2.6	50.1	1.1	2.0	0.63	25.2

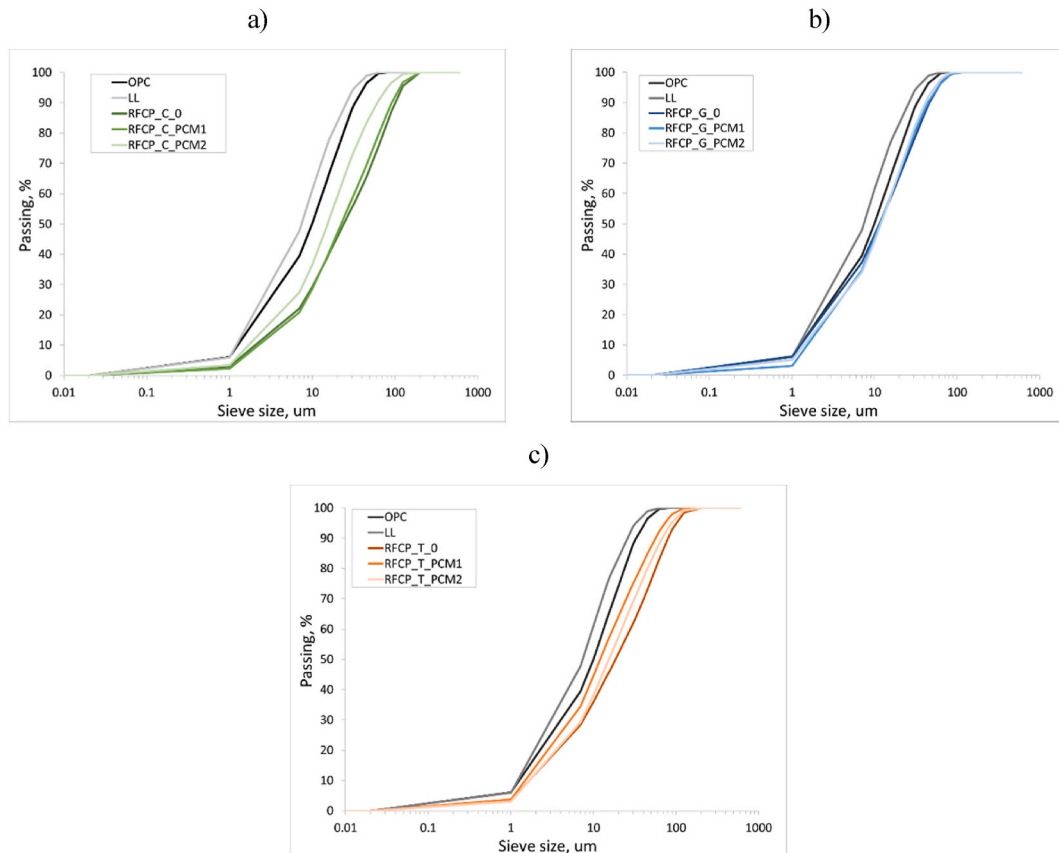


Fig. 1. Grain size distribution of OPC, LL and RFCP: a) RFCP_C; b) RFCP_G and c) RFCP_T.

small peak less noticeable [76]. The presence of MPCM significantly influences the TGA and, consequently, the DTG curves. An increased mass loss in the temperature range of approximately 200–500 °C is observed, corresponding to the evaporation of the paraffin-based MPCM. This heightened mass loss suggests that with thermal treatment of RFCP up to 750 °C, paraffin can be effectively removed from the material, indicating no anticipated negative effects of free paraffin in the mix [77,78]. It should be noted that the findings of the thermal analysis confirm the magnitude of mass loss observed during the LOI testing (Table 3), as the comparable mass loss was observed and moreover, LOI was lower for RFCP_C_0 than RFCP_C_PCM1 corresponding with the lower mass loss of RFCP_C_0 during DTA testing.

2.4. Mortars composition

Experiments were conducted on mortars with compositions following EN 196-1 [33], wherein 20 % or 40 % of the cement by weight was replaced with RFCP. The mortar compositions are detailed in Table 4. In assessing the rheological properties of mortars, those with a water-to-binder ratio (w/b) of 0.50 were found to be excessively stiff for correct measurements. Consequently, mortars with a w/b ratio of 0.65 were employed for this segment of the study.

2.5. Testing methods

The study investigated the impact of substituting a part of the cement with RFCP on various properties. This included the

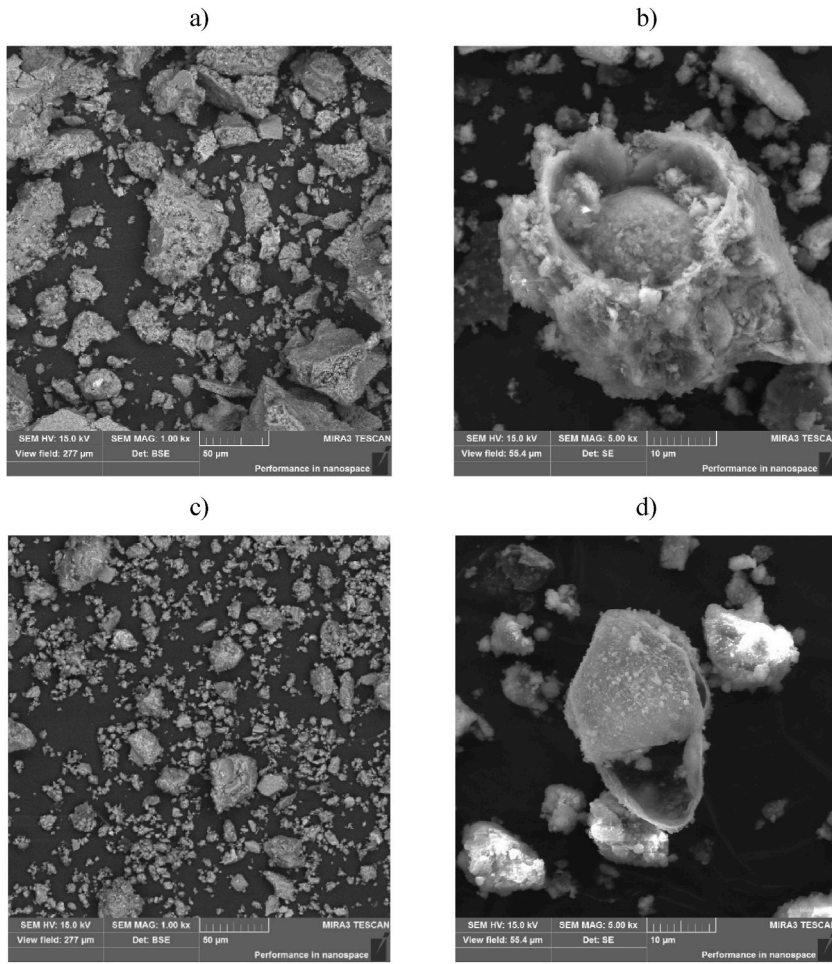


Fig. 2. SEM images: a, b) RFCP_C_PCM1 of grain size <0.125 mm (SSA 5917 cm²/g); c, d) RFCP_G_PCM1 of grain size <0.063 mm (SSA 6636 cm²/g).

fundamental properties of cement, such as the required water for standard consistency (water demand), setting time, and heat of hydration. Additionally, the research assessed the effects on fresh mortars, examining factors like consistency, rheological properties, and air content. For hardened mortars, the investigation covered properties such as flexural and compressive strength, shrinkage, water absorbability, and thermal conductivity.

The impact of RFCP on the water demand of the cement, which refers to the amount of water needed to achieve a paste with standard consistency, and on the setting time of the cement, was determined using the Vicat apparatus method outlined in EN 196-3:2016-12 [79].

The heat generated during the hydration of cement in the presence of RFCP was measured using the isothermal method specified in EN 196-11:2019-01 [80]. The isothermal calorimeter TAM Air was employed for this analysis, and the heat evolution was studied over 168 h. The paste samples consisted of 5 g of cement and RFCP, along with 2.5 g of water.

The impact of RFCP on the consistency of fresh mortar was assessed using the flow table method following EN 1015-3:2000 [81]. The rheological behaviour of mortar is commonly characterised by parameters from the Bingham model, specifically the yield stress (τ_0) and plastic viscosity (η_{pl}) [82]:

$$\tau = \tau_0 + \eta_{pl}\dot{\gamma} \quad (1)$$

where: τ – shear stress, Pa, τ_0 – yield stress, Pa, η_{pl} – plastic viscosity, Pas, $\dot{\gamma}$ – shear rate, 1/s.

Yield stress represents the stress required to initiate flow in a material, and when the shear stress surpasses the yield stress, the mix starts flowing, with its flow resistance dependent on plastic viscosity.

In this study, the Schleibinger Viskomat NT rotational rheometer was used to measure the rheological properties of mortars. The rheometer determined the yield stress parameter (g) and plastic viscosity parameter (h), corresponding to yield stress (τ_0) and plastic viscosity (η_{pl}), respectively:

$$M = g + h N \quad (2)$$

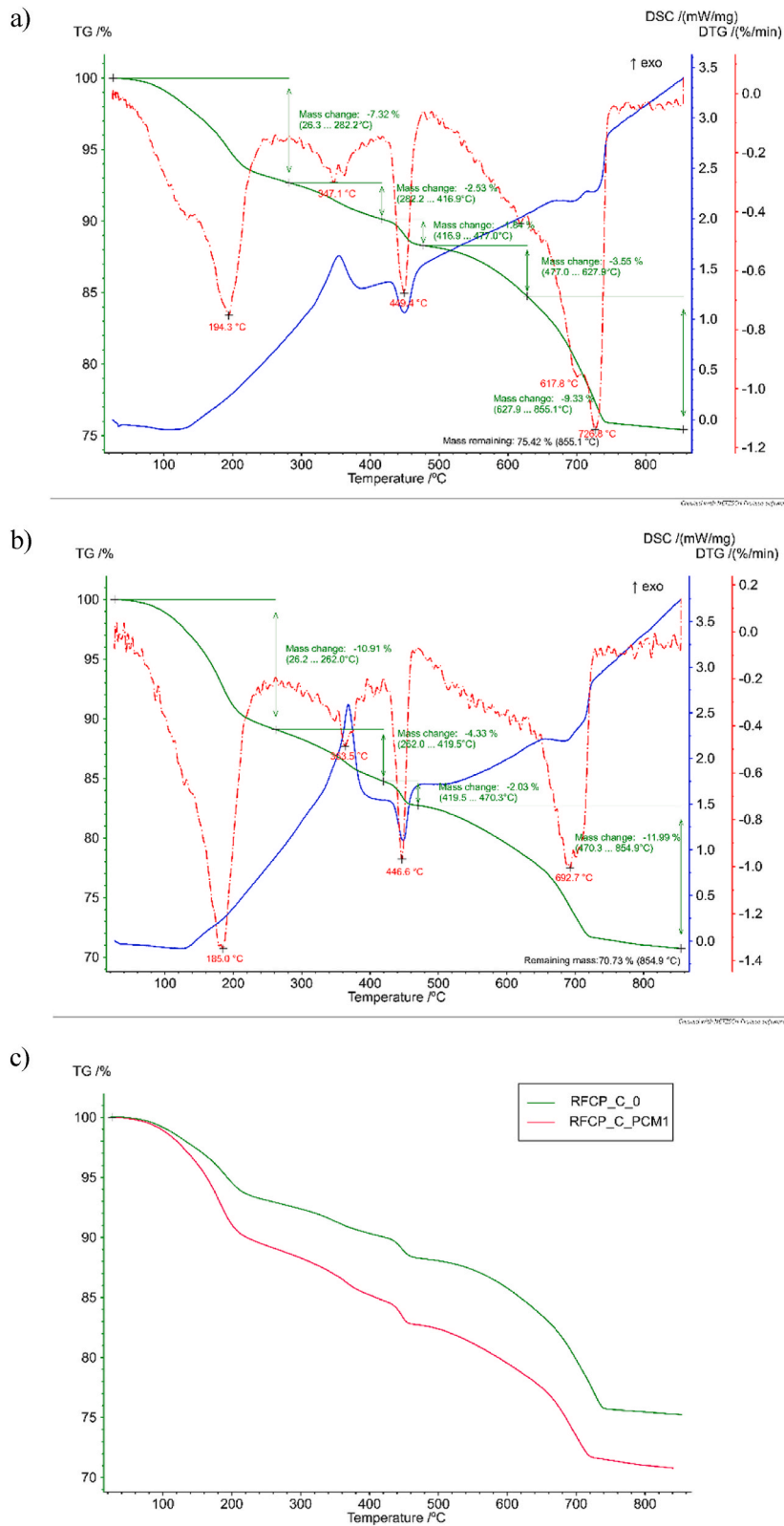


Fig. 3. Thermal analysis of the RFCP: a) RFCP_C_0 (without MPCM), b) RFCP_C_PCM1 (with MPCM Nextek 24D), c) TGA curve comparison of RFCP_C_0 and RFCP_C_PCM1.

Table 4
Mix composition of mortars [g].

Constituent	Mortar w/b = 0.50				Mortar w/b = 0.65 (used in rheological tests)			
	REF_OPC	REF_OPC_LL	20 % RFCP	40 % RFCP	REF_OPC	REF_OPC_LL	20 % RFCP	40 % RFCP
Cement (OPC)	450	360	360	270	450	360	360	270
Limestone (LL)	–	90	–	–	–	90	–	–
Water	225	225	225	225	293.2	293.2	293.2	293.2
RFCP	0	–	90	180	0	–	90	180
Sand	1350	1350	1350	1350	1350	1350	1350	1350

where: M – torque, Nmm, g – yield stress parameter, Nmm, h – plastic viscosity parameter, Nmms, N – rotational speed, 1/s. With proper calibration, these parameters can be expressed in fundamental units, as in Eq. (1). According to Ref. [83], in the apparatus used in this study, $\tau_0 = 7.9 g$ and $\eta_{pl} = 0.78 h$. Since these relationships haven't been confirmed for our specific device, finally, all results are presented in terms of yield stress parameter (g) and plastic viscosity parameter (h).

It is important to note that the rheological parameters obtained in this study range from yield stress parameter (g) of 10–90 Nmm and plastic viscosity parameter (h) from 6 to 16 Nmms. Considering the constants mentioned earlier, the yield stress (τ_0) ranges from 80 to 720 Pa, and plastic viscosity (η_{pl}) ranges from 4.7 to 12.5 Pas. These values fall within typical ranges reported in the literature for mortars [82]. The applied testing procedure was as follows. After mixing, mortar samples were transferred to the rheometer and tested after 5 and 60 min, maintaining a temperature of $20^\circ\text{C} \pm 1^\circ\text{C}$ using a thermostatic device. Next, the measurement procedure involved maintaining a constant speed of 120 rpm for 3 min, followed by measuring shear resistance at decreasing speeds of 120-100-80-60-40-30-20 rpm, with a total measurement time of 5.5 min (Fig. 4).

The impact of RFCP on the air content in fresh mortars was assessed using the pressure method as per EN 413–2:2016–11 [84].

To evaluate the effect of RFCP on flexural and compressive strengths, testing was conducted following EN 196–1:2016–07 [33] on prismatic specimens measuring 40 mm × 40 mm × 160 mm. The tests were performed after 2 and 28 days of hardening, and the reported results represent the average values derived from 3 samples (for flexural strength) and 6 samples (for compressive strength).

The influence of RFCP on the water absorption of mortar was determined on 40 mm × 40 mm × 160 mm prismatic specimens after 28 days of hardening. The specimens underwent a process of drying to a constant mass at $105^\circ\text{C} \pm 5^\circ\text{C}$ until a constant mass was achieved. The water absorption, W_a , was calculated as the ratio of water mass absorbed to that of the dry mass of the sample and expressed as a percentage:

$$W_a = 100 \frac{(m_w - m_d)}{m_d} \quad (3)$$

where m_w is the mass of the wet sample, g, m_d is the mass of the dried sample, g.

The impact of RFCP on mortar shrinkage was measured using 40 mm × 40 mm × 160 mm prismatic specimens, employing the Graf-Kaufman method over 1–29 days. The samples were stored in a climate chamber at 20°C and 60 % relative humidity, and the reported results represent the average of the three samples.

For the thermal properties of mortars, including thermal conductivity and volume heat capacity, assessments were conducted on 100 mm cubes after 28 days of curing in water at 20°C . The thermal properties were measured after drying the specimens to a constant mass at $105^\circ\text{C} \pm 5^\circ\text{C}$. The thermal analyser Isomet 2114 [85] was used, employing a dynamic method based on analysing the material's temperature response and heat flow impulses. The reported results represent the average of three measurements.

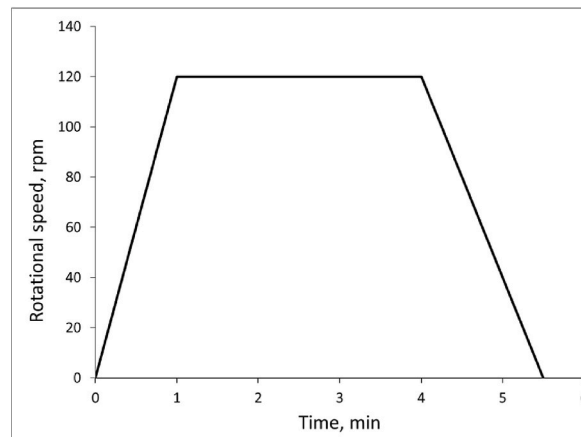


Fig. 4. The measurement procedure used in the rotational rheometer.

3. Results and discussion

3.1. Water required for standard consistency of pastes

The impact of the type and quantity of RFPC on the necessary water for achieving the standard consistency of cement pastes is illustrated in Fig. 5.

The required water for achieving the standard consistency of cement pastes, both OPC and OPC_LL, is approximately 29 %. Introducing 20 % or 40 % RFPC, regardless of the RFPC type, leads to an increased water requirement to attain the standard consistency paste. Consequently, the water demand of RFPC surpasses that of OPC and LL. For RFPC_C and RFPC_G, the required water for standard consistency shows a linear increase with the rising addition of RFPC from 20 % to 40 %, ranging from 4 % (RFPC_G_PCM1) to even 10 % (RFPC_C_PCM2). Notably, the water required for standard consistency in cement paste with RFPC_G is lower than that with RFPC_C, especially at higher RFPC additions. The addition of 20 % heat-processed RFPC_T needs the water for standard consistency of cement pastes at 36 %–42 %, thus values higher than that obtained for OPC and OPC_LL (29 %). Difficulties in achieving a stable standard consistency with 40 % RFPC_T resulted in the water requirement not being determined for those samples.

Based on the water demand of the pastes with RFPC, the water demand of the RFPCs was calculated. It should be noted, that while the water absorption is an important parameter of the recycled aggregate and can be easily calculated for aggregate grains [86], there is a difficulty in providing the same measurements for the fine particles of the used RFPC. RFPC has a high specific surface area, higher than OPC used in the testing, and therefore it is hard to distinguish whether water kept by the RFPC was absorbed in the material, or in which part adsorbed on its surface or between the grains due to capillary effects. Therefore, rather than considering water absorption, as is the case with recycled aggregate, calculating water demand can serve as a parameter to predict its impact on consistency and workability.

The water demand of RFPC, calculated based on the standard consistency, is shown in Table 5.

The influence of the processing method of RFPC without MPCM on the water required for the standard consistency of cement pastes and water demand can be attributed to several factors. Firstly, the hydrated cement in RFPC exhibits a higher specific surface area compared to unhydrated cement [75], resulting in an elevated water demand. Additionally, RFPC_C_0, characterized by the presence of relatively large, porous grains with a developed internal surface, further increases water demand. Grinding the large grains of RFPC_C should theoretically reduce water demand in RFPC_G. However, this is offset by an increase in specific surface area during grinding, leading to similar water demands for RFPC_G_0 and RFPC_C_0. In the case of RFPC_T_0, heat treatment causes CH and C–S–H to dehydrate, creating micro-cracks in the structure [19]. This results in an increased specific surface area and water demand compared to non-thermally processed RFPCs. Moreover, the dehydrated products react with water immediately after mixing, further escalating the water demand of RFPC_T_0, making it the treatment method with the highest water demand in comparison to others.

The impact of MPCM presence in RFPCs on water demand varies depending on the treatment method. Specifically, the inclusion of MPCM in RFPC_C leads to a notable rise in the water needed to achieve standard consistency in cement paste—approximately 4–7% water higher than in RFPC_C_0. This heightened water demand in RFPC_C with MPCM can be attributed to the formation of RFPC agglomerates, primarily caused by lumps of paraffin released from the capsules, as illustrated in Fig. 2. The grinding process reveals that the impact of MPCM presence in RFPC on the required water for standard consistency becomes noticeable only when 40 % RFPC_G is added, and even then, it is insignificant. It is possible that the grinding process contributes to a more even distribution of paraffin on the surface of RFPC grains. The combination of a relatively small amount of paraffin and the increased fineness of RFPC_G results in a diminished agglomeration effect. This observation is supported by the Laser Diffraction Analysis (LDA) depicted in Fig. 1. Notably, RFPC_C with MPCM exhibits significantly larger grain sizes compared to RFPC_C_0, while the grain size of RFPC_G shows less dependence on the presence of MPCM. However, further research is needed to explore this issue, along with clarifying the influence of MPCM type on the water demand of RFPCs. The impact of MPCM type on the required water becomes apparent with the addition of 40 % RFPC_G, whereas pastes with RCPM_G_PCM1 require less water for standard consistency.

Surprisingly, the origin of RFPC_T has an impact on the necessary water content. Cement pastes obtained from ULFCs with MPCM,

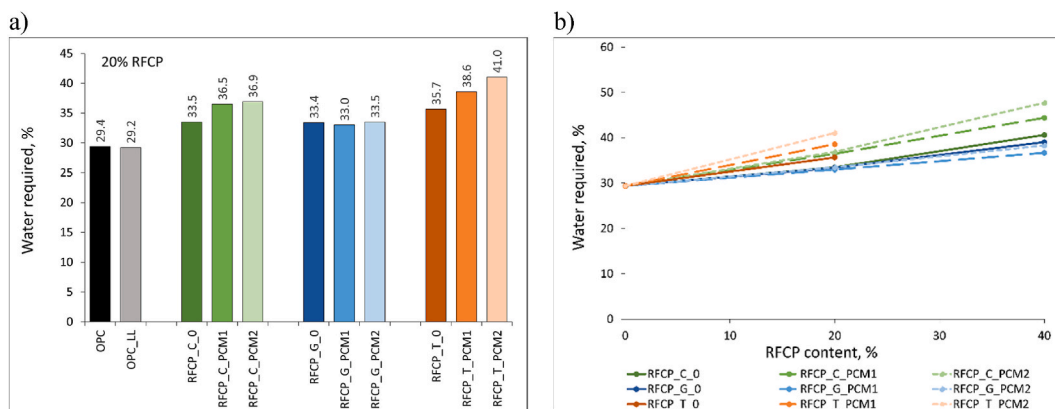


Fig. 5. Influence of type (a) and content of RFPC (b) on water required for standard consistency of cement pastes.

Table 5
Water demand of RFCPs used in the research.

RFCP type	Water demand [dm ³ /kg]
RFCP_C	53.6
RFCP_C_PCM1	65.9
RFCP_C_PCM2	71.0
RFCP_G	51.4
RFCP_G_PCM1	47.6
RFCP_G_PCM2	50.8
RFCP_T	60.8
RFCP_T_PCM1	75.3
RFCP_T_PCM2	87.6

specifically RFCP_T_PCM1 and RFCP_T_PCM2, demand more water for standard consistency compared to pastes with RFCP_T.0. Additionally, the influence of the MPCM type is distinctly observable. The evaporation and/or combustion of MPCMs during thermal treatment may potentially enhance the porosity of the RFCP microstructure, but this matter necessitates further investigation.

3.2. Initial setting time of pastes

Fig. 6 illustrates the initial setting time of pastes with standard consistency, both with and without RFCP. Generally, the addition of RFCP delays the initial setting time of cement pastes, with a notable impact from the type and, in the case of RFCP_C, the quantity of RFCP. Specifically, the inclusion of 20 % RFCP_C_0 and RFCP_G_0 (without MPCM) results in a delay of approximately 1.6 and 1.9 h respectively compared to OPC paste and about 1 h compared to OPC_LL paste. Therefore, the grinding process applied to RFCP does not significantly alter the initial setting time. Further increasing the amount of RFCP_C_0 to 40 % leads to a linear extension of the setting time delay. In contrast, the quantity of RFCP_G_0 has no significant impact on the initial setting time. Pastes with RFCP_T_0 exhibit a faster initial setting time than those with other RFCPs but are still delayed compared to OPC paste. The delay in the initial setting time results from the substitution of some cement with inactive RFCP_C or RFCP_G, or less chemically active than cement RFCP_T, thereby diluting the concentration of cement grains. It is important to note that while limestone powder is considered to be mostly chemically inactive, its fine grains (usually much finer than cement grains) can serve as nuclei for crystallization, and depending on its content can provide a smaller delay, or even an acceleration of the initial setting. This effect on hydration, and thus setting, was widely reported in existing literature [87–91]. Limestone particles provide very effective nucleation sites due to their shape, and easy adsorption of calcium ions on their surface, which results in stable CSH phase nuclei and thus increased hydration rates [92–95]. Limestone particles were proven to be a more effective nucleation sites, and thus provide a better increase in early hydration speed, than fillers like quartz, slag, fly ash, or cement particles [92–94]. While RFCP can also be considered in light of additional nucleation sites, due to their large specific surface area, it may not provide the same benefits as limestone, while also containing several substances that may inhibit hydration such as a foaming agent [96]. This explains the relatively lower delay in the setting time of OPC_LL paste compared to pastes with RFCP. The lower impact on the delay in the initial setting time of RFCP_T is attributed to its partial activity.

The presence of MPCM in RFCP_C significantly prolongs the initial setting time of pastes, causing delays ranging from approximately 1.5 h to about 3 h compared to pastes with RFCP_C_0 (at 20 % and 40 % RFCP addition, respectively). In the case of RFCP_G, the impact of MPCM presence on the initial setting time delay is less pronounced, not exceeding 0.5 h when compared to pastes with RFCP_G_0. The delay attributed to MPCM presence is likely due to the free paraffin released from the capsules promoting the agglomeration of cement grains and obstructing water access to the cement surface. This effect is particularly evident in RFCP_C, which contains clumps of free paraffin. In contrast, the grinding process in RFCP_G ensures that paraffin is bound to the surface of the RFCP grains, minimizing its impact on cement agglomeration and water access. Interestingly, even though MPCM has been removed from RFCP_T, the delay in the initial setting time of pastes with RFCP_T sourced from ULFC with MPCM remains visible. This effect may be

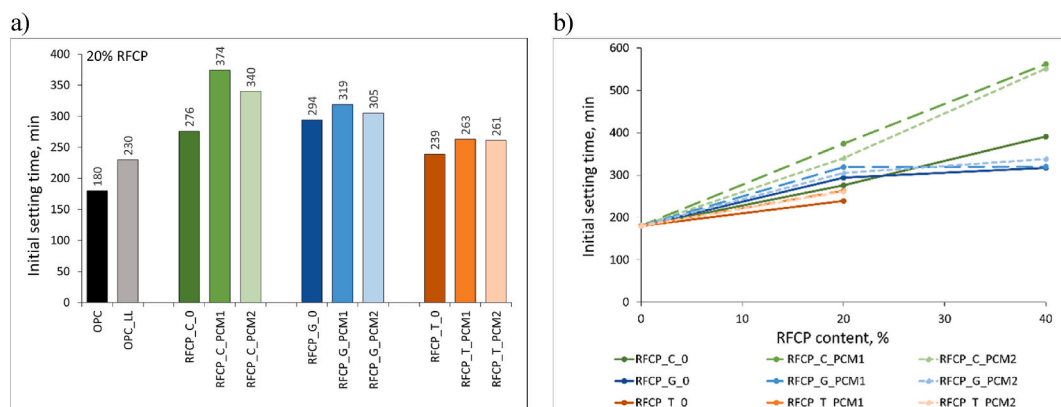


Fig. 6. Influence of type (a) and content of RFCP (b) on the initial setting time of pastes with standard consistency.

attributed to the need to add more water to achieve the required consistency in cement pastes with RFCP_T_PCM1 and RFCP_T_PCM2 compared to cement paste with RFCP_T_0 (see Section 3.1).

3.3. Hydration heat of cement

Figs. 7–8, along with Table 6, illustrate the impact of adding RFCP as a 20 % cement replacement on hydration heat evolution curves and the rate of heat evolution of pastes. In general, regardless of the type and source of RFCP, its addition results in a reduction of both the rate of heat evolution and the total heat evolved during the hydration process. The addition of RFCP extends the induction and acceleration period of cement hydration, aligning with the findings on initial setting times presented in Section 3.2. Further, a main hydration peak is delayed and diminished. RFCP_C_0 and RFCP_G_0 exhibit a more pronounced effect on cement hydration heat compared to RFCP_T_0. The relatively lower impact of RFCP_T_0 on the evolved heat, compared to other RFCPs, can be attributed to its reactivity, albeit lower than that of cement. Initially, the heat evolved by OPC_LL is higher than that of cement with RFCP_C_0 and RFCP_G_0. This discrepancy is because the finely ground OPC_LL acts as a nucleus for crystallization, a function not fully fulfilled by the grains of RFCP [89]. High hydration heat in the early stages of hydration of cements with limestone due to the nucleation effect was previously reported in existing literature [90,97–99]. As mentioned before, limestone filler can have beneficial effects on early hydration due to its lattice cleavage [92] and its ability to engage in ion exchange with cement particles [92–94]. However, after 24 h, the total heat evolved by cement with RFCP_C_0 and RFCP_G_0 becomes comparable to that of OPC_LL. This occurs because although limestone presence can increase the initial hydration rate, it's essentially inert and doesn't contribute to extra heat production. Consequently, in the long term, the dilution effect remains consistent, similar to RFCP_C_0 and RFCP_G_0 [90].

The impact of RFCP_C_0 and RFCP_G_0 on cement hydration is comparable, as their presence delays the end of induction and the occurrence of the main peak by approximately 1.5 h compared to OPC. Surprisingly, though, the addition of RFCP_G_0 results in a greater reduction in heat release compared to the addition of RFCP_C_0. Specifically, the main hydration peak of paste with RFCP_C_0 or RFCP_G_0 is reduced by 20 % and 33 %, respectively, in comparison to OPC paste. The inclusion of RFCP_C_0 or RFCP_G_0 leads to a

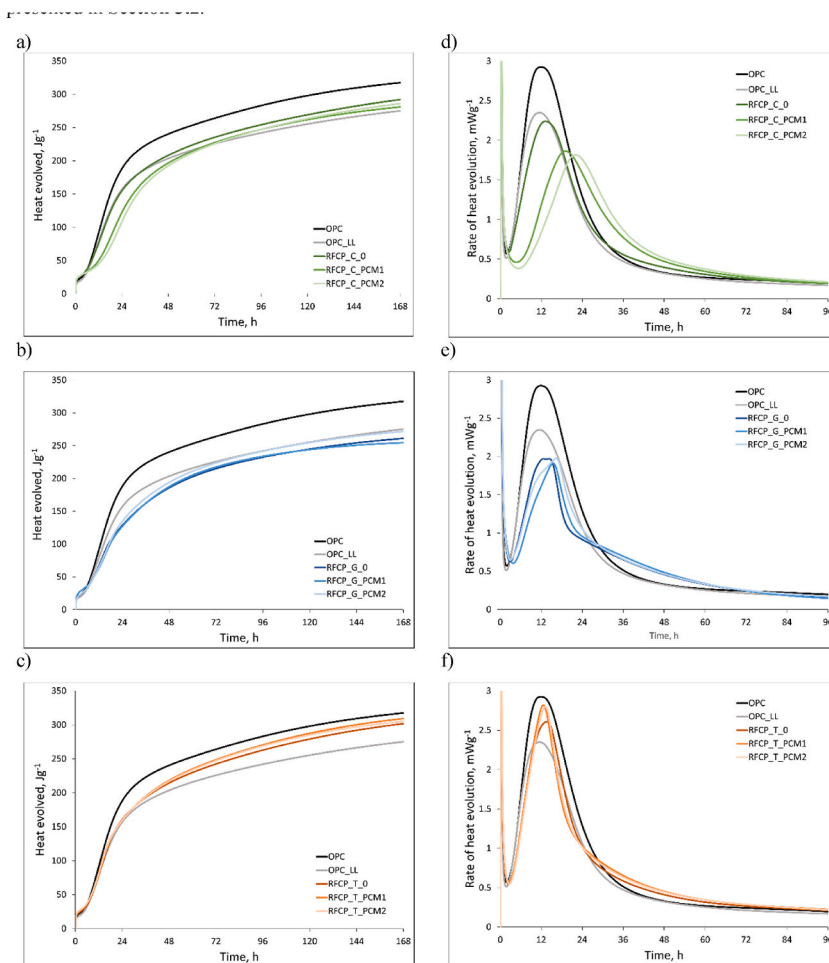


Fig. 7. The heat evolved in the presence of 20 % RFCP as cement replacement: a) RFCP_C, b) RFCP_G, c) RFCP_T and the rate of the heat of evolution of pastes with 20 % RFCP: d) RFCP_C, e) RFCP_G, f) RFCP_T.

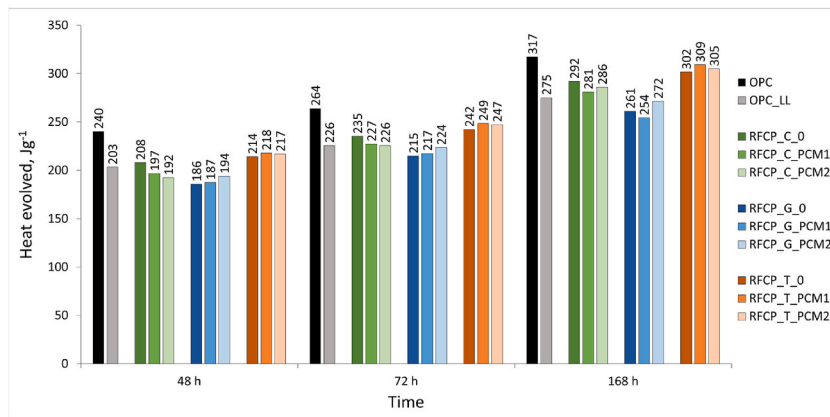


Fig. 8. The heat evolved in the presence of 20 % RFCP as cement replacement.

Table 6

Key values of the heat of hydration of cement pastes without and with RFCP.

Paste	End of the induction period, h	Time of the main peak, h	The main peak of heat evolution rate, mWg ⁻¹	Heat evolved related to OPC paste, %		
				48 h	72 h	168 h
OPC	2.8	12.0	2.92	100.0	100.0	100.0
OPC_LL	3.2	11.5	2.35	84.7	85.5	86.7
RFCP_C_0	3.9	13.3	2.34	86.6	89.1	92.0
RFCP_C_PCM1	7.5	19.2	1.86	81.8	86.1	88.5
RFCP_C_PCM2	6.5	22.1	1.85	80.1	85.5	90.1
RFCP_G_0	4.5	12.6	1.97	77.3	81.4	82.3
RFCP_G_PCM1	5.0	15.5	1.91	78.0	82.4	80.2
RFCP_G_PCM2	4.5	16.4	1.98	80.7	84.7	85.6
RFCP_T_0	3.5	13.7	2.61	89.2	91.8	95.1
RFCP_T_PCM1	3.5	12.6	2.82	90.8	94.2	97.4
RFCP_T_PCM2	3.5	13.2	2.80	90.3	93.7	96.1

decrease in evolved heat after 48 h relative to OPC by approximately 15 % and 23 %, and after 168 h by approximately 8 % and 18 %, respectively. One possible explanation for the grinding effect is that the finer grains of RFCP_G_0 may contribute more than RFCP_C_0 to cement agglomeration, impeding water access to the cement and thereby reducing heat evolution during the initial hydration period. However, further studies are needed to validate this effect. Conversely, the introduction of RFCP_T_0 only slightly delays the end of the induction period and the main peak occurrence (by about 1 h), reduces the main hydration peak (by about 10 %) and decreases the evolved heat after 168 h (by about 5 %). Overall, the influence of RFCP_T_0 on cement hydration heat determined in this study aligns with findings from previous research [19].

The impact of MPCM presence in RFCP on cement hydration is notably pronounced, particularly in the case of RFCP_C. The conclusion drawn is that for paste with RFCP_C_PCM1 or RFCP_C_PCM2, the end of induction is significantly delayed by about 4 h relative to RFCP_C_0, with even more substantial delays of 6–9 h for the main hydration peak. The main hydration peak values are also 20 % lower, and the total heat evolved after 24 h is approximately 30 % lower. Notably, the type of MPCM influences this effect, with RFCP_C_PCM2 causing a more significant delay in the main peak occurrence compared to RFCP_C_PCM1, as well as resulting in less heat evolved after 24 h. Since the rate of heat evolution of paste with RFCP_C_PCM1 or RFCP_C_PCM2 between 20 and 70 h is higher than that with RFCP_C_0, this partially offsets the initially reduced heat evolution. Thus, after 168 h, the total heat evolved by paste with RFCP_C_PCM1 or RFCP_C_PCM2 is insignificantly lower than that in the presence of RFCP_C_0 (by 2–3%). The influence of the MPCM type in RFCP_C is particularly significant in the first 24 h of cement hydration, disappearing completely thereafter. In contrast, for cement with RFCP_G, the presence of MPCM has a minor effect on the hydration process. The end of the induction period for pastes with RFCP_G_PCM1 or RFCP_G_PCM2 relative to RFCP_G_0 is similar, and although the main hydration peak is delayed by 3–4 h, the main hydration peak value remains the same. The effect of MPCM in RFCP_G on total heat evolution up to 168 h is insignificant. The influence of adding RFCP_T_PCM1 and RFCP_T_PCM2 on heat evolution and the rate of heat evolution of pastes is analogous to the effect of adding RFCP_T_0. In conclusion, the presence of MPCM in RFCP affects the hydration heat during the early hydration period, up to 24 h. These findings align with the results of the research on the effect of RFCP on the setting time (see Section 3.2) and the duration of cement hydration periods. The mechanism of the effect of RFCP appears to be analogous to setting time as presented in Section 3.2.

3.4. Consistency and rheological properties of fresh mortars

The influence of RFCP type and content on the consistency of fresh mortars with water-to-binder ratios (w/b) of 0.50 and 0.65 is depicted in Figs. 9–10. Additionally, Figs. 11–12 illustrate the rheological properties of mortars tested with a w/b of 0.65. However, the high water demand of RFCP_C and RFCP_T resulted in fresh mortars containing 40 % of these RFCPs being excessively dry, rendering them unsuitable for consistency and rheological tests.

The addition of 20 % RFCP, regardless of its type, leads to a reduction in mortar flow, with the impact being least pronounced with RFCP_G and most significant with RFCP_T (Fig. 9a). The presence of MPCM in RFCP_C (RFCP_C_PCM1 and RFCP_C_PCM2) negatively influences the consistency of mortars, distinctly decreasing their flow compared to mortars with RFCP_C_0. Further, the presence of MPCM does not affect the consistency of mortars with 20 % RFCP_G - the flow of mortars with RFCP_G_0, RFCP_G_PCM1, and RFCP_G_PCM2 shows no significant differences. Also, mortars with RFCP_T with MPCM exhibit lower flow than mortars with RFCP_T_0. The type of MPCM does not impact the consistency of mortars with 20 % RFCP_C or RFCP_G. At 40 % RFCP_G addition, the flow of mortars with RFCP_G_PCM1 and RFCP_G_PCM2 is higher than that of mortars with RFCP_G_0, and mortars with RFCP_G_PCM1 exhibit a slightly higher flow rate than those with RFCP_G_PCM2 (Fig. 9b). On the contrary mortars with RFCP_T with MPCM exhibit slightly lower flow than mortars with RFCP_T_0. The flow of mortars, both with and without RFCP, decreases with time. The flow loss over time for mortars with processed powders RFCP_G and RFCP_T is insignificant and unrelated to the presence of MPCM in them. For mortars with RFCP_C, the presence of MPCM increases the flow loss over time (Fig. 10).

A flow-table test provides limited information on the rheological properties of mortar, as the sample undergoes cyclic dynamic loads, transitioning into a fluid state, and only one parameter, the flow diameter, is measured. To obtain a more comprehensive understanding, measurements were conducted using a rheometer, and the results are presented in Figs. 11–12. The graphs depict the yield stress parameter (g) and plastic viscosity parameter (h), corresponding to the yield stress (τ_0) and plastic viscosity (η_{pl}), as defined in Eq. (2) and Eq. (1), respectively (see Section 2.5 for details).

The addition of 20 % RFCP enhances both the yield stress (g) and plastic viscosity (h) of the mortar. The magnitude of this enhancement varies depending on the type of RFCP used. For mortars with RFCP_T, the increase in yield stress (g) is the most significant, ranging from approximately 300 %–400 % compared to OPC and OPC_LL mortars (Fig. 11). Following this, mortars with RFCP_C exhibit an increase of approximately 30 %–40 %, while mortars with RFCP_G show a more modest increase of about 10 %. As for plastic viscosity (h), mortars with RFCP_C experience the highest increase, ranging from approximately 100 %–170 % compared to OPC and OPC_LL mortars (Fig. 12). Mortars with RFCP_T follow closely with an increase of approximately 120 %–150 %, and mortars with RFCP_G exhibit a more modest increase of around 30 %. In mortars without RFCP (OPC and OPC_LL), the yield stress (g) increases by approximately 5 Nmm over 60 min. The presence of RFCP accelerates this increase in yield stress (g) over time, with the most significant impact observed in mortars with RFCP_T (up to 27 Nmm), followed by RFCP_C (up to 15 Nmm) and RFCP_G (up to 10 Nmm). After 60 min, the plastic viscosity (h) of all mortars is up to 2 Nmms higher than the initial values. However, these changes have a minor influence on the flowability of the mortars.

At a 40 % addition of RFCP, the measurement of rheological parameters was feasible only for mortars containing RFCP_G. Elevating the RFCP_G addition from 20 % to 40 % leads to a substantial increase in the yield stress (g) ranging from 160 % to 230 %. In comparison to Ordinary Portland Cement (OPC) mortar, the yield stress (g) experiences an up to 350 % increase, while the plastic viscosity (h) shows an increase from 120 % to 160 %. The increase in yield stress (g) for mortars with 40 % RFCP_G over a 60 min reaches up to 15 Nmm, mirroring the behaviour observed in mortars with 20 % RFCP_G. Notably, the plastic viscosity (h) of mortars with 40 % RFCP_G remains relatively stable over time.

The impact of MPCM presence in RFCP on the rheological properties of mortars depends on the processing method employed. In the case of mortars with RFCP_C, the inclusion of MPCM (RFCP_C_PCM1 and RFCP_C_PCM2) increases both yield stress (g) and plastic viscosity (h). Conversely, the introduction of 20 % RFCP_G with PCM (RFCP_G_PCM1 and RFCP_G_PCM2) does not alter the rheological properties of the mortars. Surprisingly, at a 40 % RFCP_G addition, the yield stress (g) and plastic viscosity (h) of mortars with

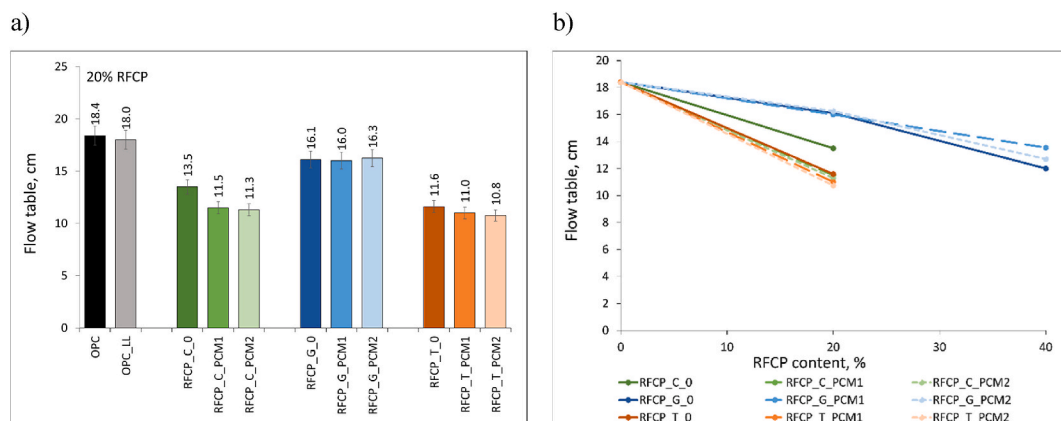


Fig. 9. Influence of type (a) and content (b) of RFCP on the consistency of fresh mortars with w/b = 0.50.

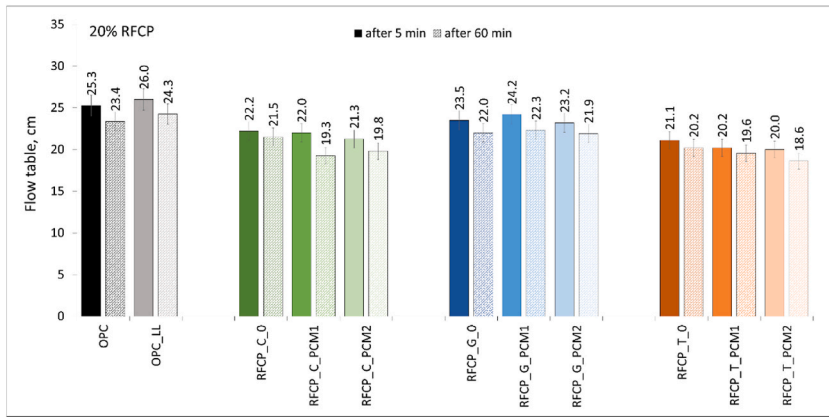


Fig. 10. Influence of type and content of RFPC on the consistency of fresh mortars with w/b = 0.65 after 5 and 60 min.

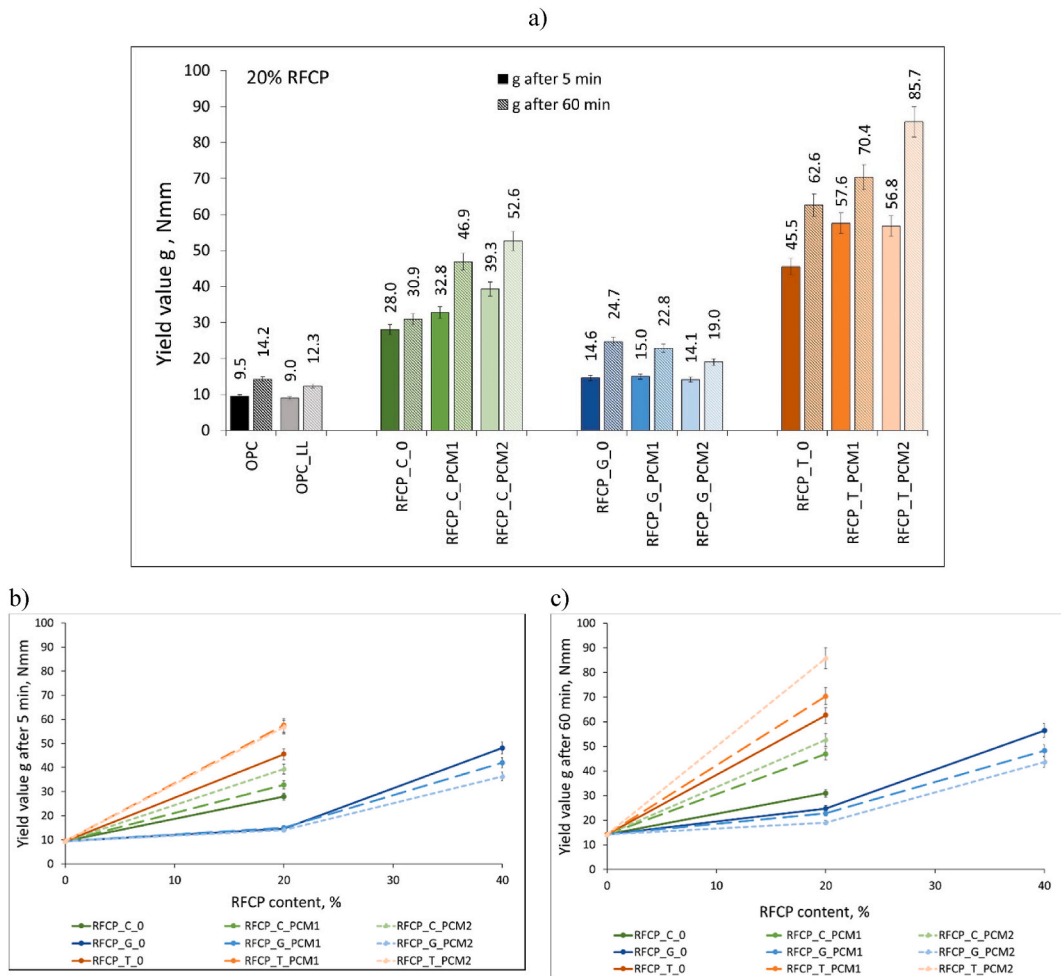


Fig. 11. Influence of type (a) and content of RFPC on yield stress (g) of fresh mortars with w/b = 0.65 after 5 min (b) and 60 min (c).

RFPC_G_PCM1 and RFPC_G_PCM2 are lower than those with RFPC_G.0. On the other hand, the yield stress (g) and plastic viscosity (h) of mortars with RFPC_T obtained from ULFC with MPCM (RFPC_T_PCM1 and RFPC_T_PCM2) surpass those of mortars with RFPC_T.0. The presence of MPCM in RFPC accelerates the increase in yield stress (g) over time in mortars with RFPC_C but decelerates the increase in yield stress (g) of mortars with RFPC_G. The increase in yield stress (g) over time in mortars with RFPC_T sourced from ULFC

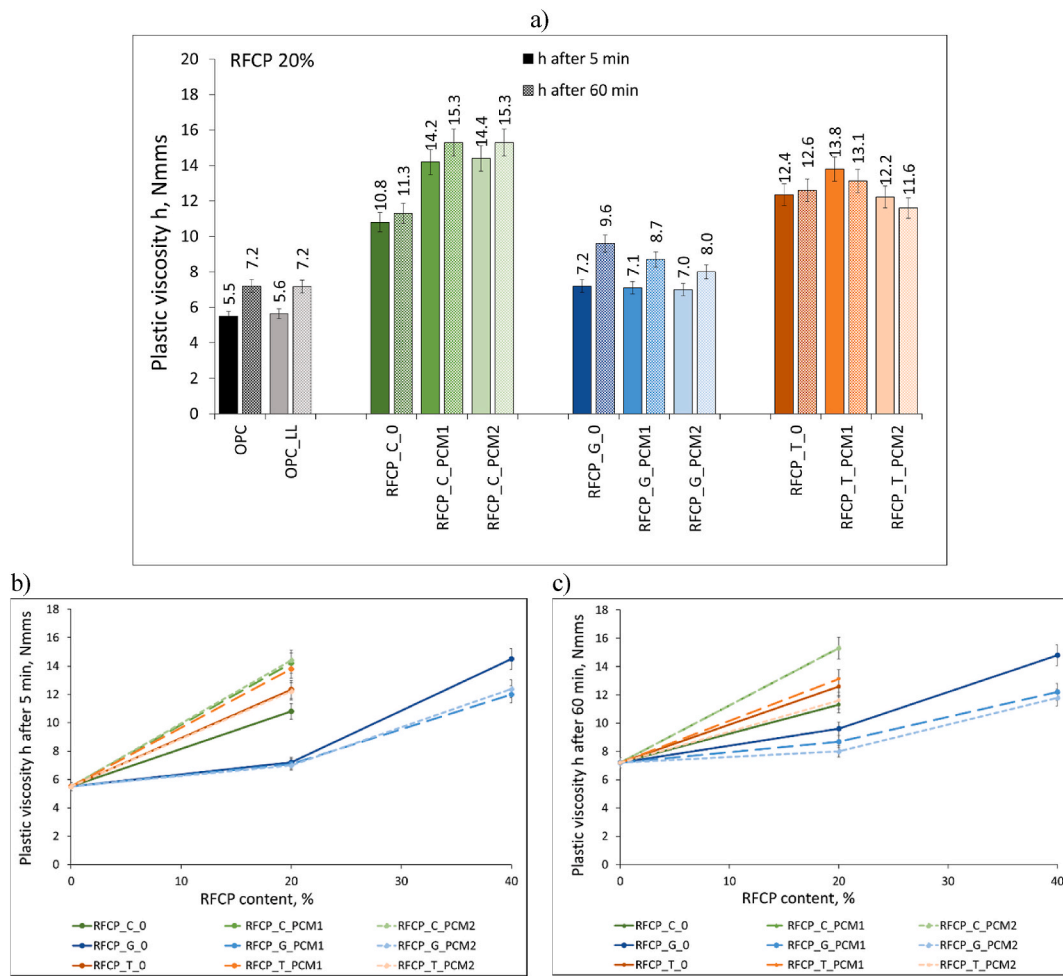


Fig. 12. Influence of type (a) and content of RFPC on plastic viscosity (h) of fresh mortars with $w/b = 0.65$ after 5 min (b) and 60 min (c).

with MPCM is faster than those with RFPC_G.0. Changes in plastic viscosity (h) over time for both reference and RFPC-infused mortars exhibit a low range and are essentially unrelated to the type of RFPC incorporated into the mortar. At a 20 % RFPC addition with MPCM, the influence of MPCM type on the consistency and rheological properties of mortars is negligible. However, with a 40 % RFPC addition, mortar with RFPC_G_PCM2 demonstrates higher flowability.

Further, Fig. 13 illustrates the correlation between the flow and yield stress (g) of the mortars investigated in this study. This

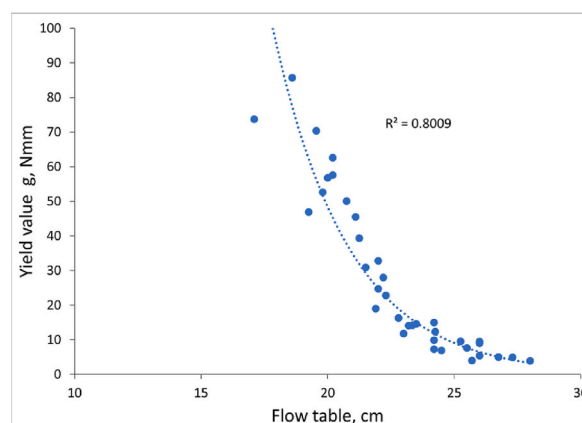


Fig. 13. Relationship between flow table diameter and yield stress (g) of fresh mortars with RFPC addition.

relationship confirms the well-documented possibility, as found in the literature (e.g., Refs. [100,101]), of determining the yield stress (g) of mortars through flow table measurements. However, direct measurement of the yield stress (g) using a rheometer provides a significantly enhanced understanding of the impact of material and technological factors on the rheological properties of the mortar. This is evident in Fig. 14, where it is apparent that rheometer measurements offer higher resolution compared to flow table measurements, showing more clearly the differences between mortar consistency.

The influence of RFCP on the consistency and rheological properties of mortars, as depicted in Fig. 14, depends on the water demand of the RFCP. A higher water demand corresponds to reduced flow and elevated yield stress (g) and plastic viscosity (h) in the fresh mortar. The variation in mortar consistency due to RFCP processing and the presence of MPCM in RFCP stems from their influence on RFCP water demand, as detailed in Section 3.1. Additionally, the size and shape of RFCP grains influence mortar rheological properties. RFCP_C and RFCP_T grains are relatively large, exceeding the size of cement grains, and possess an irregular shape with a rough surface, resulting in an increase in both yield stress (g) and plastic viscosity (h) upon their addition to the mortar. In contrast, processing through grinding renders RFCP_G grains finer, exhibiting a regular spherical shape and a relatively smooth surface. Consequently, mortars with RFCP_G experience a lower increase in yield stress (g) and plastic viscosity (h) compared to mortars with other types of RFCPs.

3.5. Air content in fresh mortars

Fig. 15 illustrates the impact of both the type and quantity of RFCP on the air content in fresh mortars with a water-to-binder ratio (w/b) of 0.50. The addition of 20 % RFCP_C_0, RFCP_G_0, or RFCP_T_0 results in an approximately 0.5 %–1.5 % increase in the mortar's air content, respectively, when compared to Ordinary Portland Cement (OPC) mortar. It is noteworthy that the addition of RFCP to the mortar contrasts with the effect of adding limestone powder LL, which typically reduces the air content in the mortar. The presence of MPCM in both RFCP_C and RFCP_G leads to a reduction in the mortar's air content by approximately from 1 % to 2 %, bringing it to a level comparable to OPC mortar. Increasing the quantity of RFCP_G_0 does not affect the air content of the mortar, whereas an increase in the amount of RFCP_G with MPCM results in a 1 % increase in air content. In general, the addition of RFCP significantly raises the air content in mortars, but the inclusion of MPCM in RFCP has a mitigating effect, reducing the overall air content in the mortar.

3.6. Density of hardened mortars

The impact of both the type and quantity of Recycled Fine Ceramic Particles (RFCP) on the density of hardened mortars with a water-to-binder ratio (w/b) of 0.50 is shown in Fig. 16. The density of hardened mortars with 20 % RFCP is approximately 3 %–5.8 % lower than that of OPC mortars. The type of RFCP and the presence of MPCM in the RFCP have an insignificant effect on the density of the mortars, as the density of all mortars with RFCP falls within the range of 2.01 to 1.95 g/cm³. Notably, increasing the amount of RFCP_G to 40 % results in a reduction in density by approximately 2.5–3.5 %.

3.7. Flexural and compressive strength

Figs. 17–18 depict the influence of RFCP type and content on the flexural strength and compressive strength of mortars after 2 and 28 days, respectively while Table 7 presents the development of flexural and compressive strength in RFCP mortars related to the values determined for OPC mortar.

The addition of 20 % RFCP significantly diminishes the 2-day flexural strength of the mortars Fig. 17a–Table 7). Specifically, the flexural strength of RFCP_C and RFCP_G mortars is 30 %–35 % lower than OPC mortar and 15 %–25 % lower than OPC_LL mortar. Mortars with RFCP_T exhibit a 10 %–13 % decrease in flexural strength compared to OPC mortar, which is similar to OPC_LL mortar, and a 25 %–35 % increase compared to mortars with RFCP_C and RFCP_G. Notably, mortars with RFCP_C_0 and RFCP_G_0 exhibit no significant difference in flexural strength. The presence and type of MPCM in RFCP_C and RFCP_G do not impact the flexural strength of

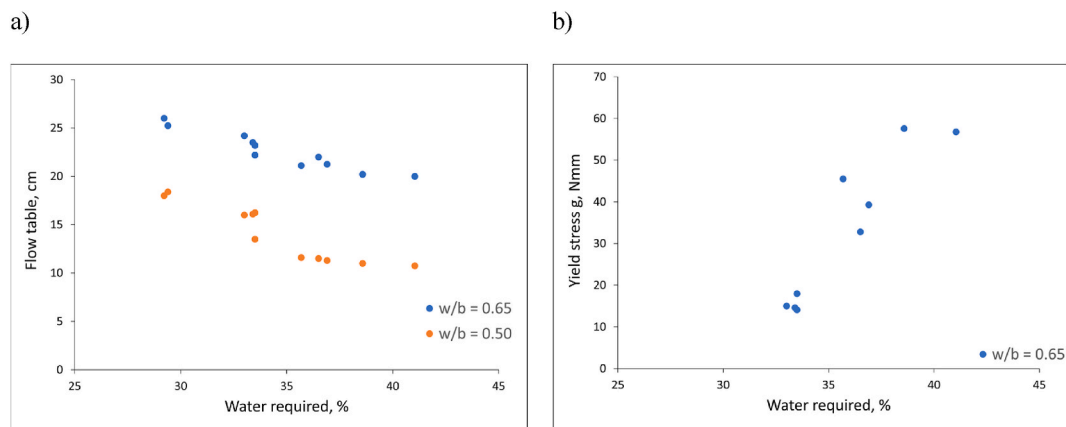


Fig. 14. Relationship between water required for standard consistency of paste and: a) flow of mortars of different w/b ratios, b) yield stress (g) of mortars.

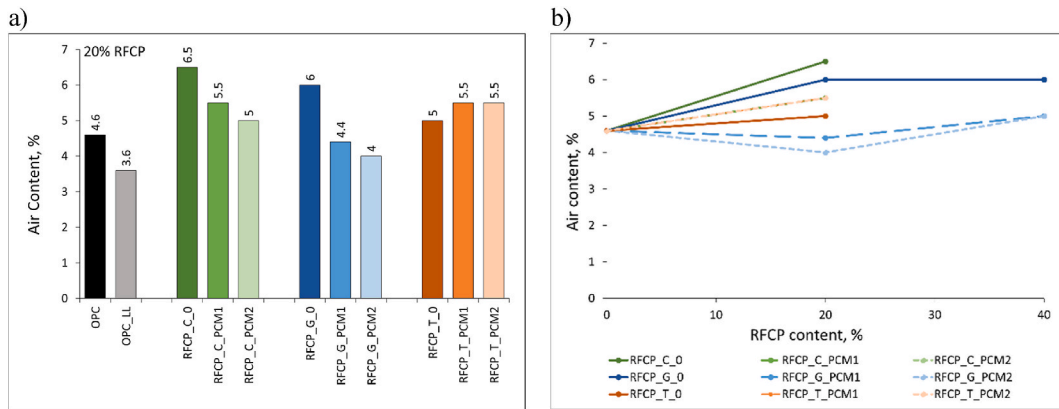


Fig. 15. Influence of type (a) and content (b) of RFCP on air content in fresh mortars with $w/b = 0.50$.

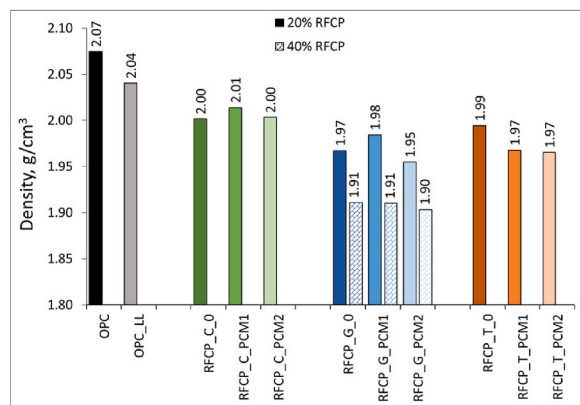


Fig. 16. Influence of RFCP type and content on the density of hardened mortars.

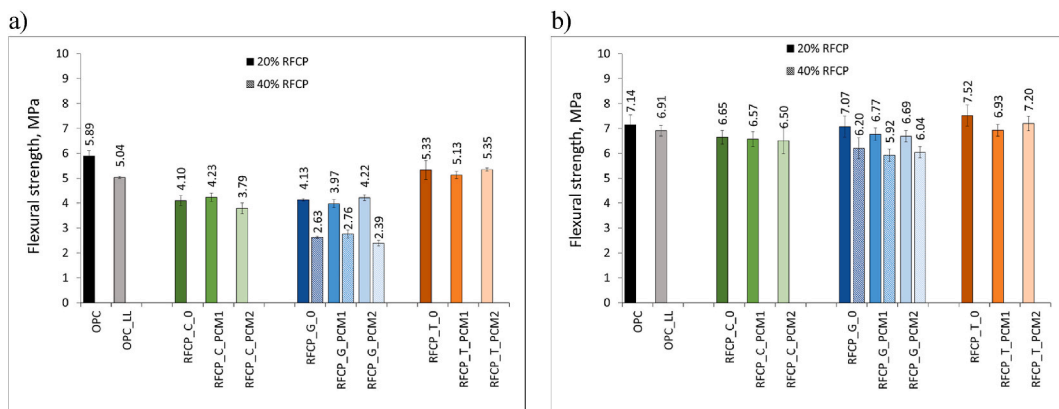


Fig. 17. Influence of RFCP type and content on flexural strength of mortars: a) after 2 days, b) after 28 days.

the mortars. Additionally, the source of RFCP_T, whether from ULFC without or with MPCM, does not influence the flexural strength of mortars.

After 28 days, the effect of RFCP on the flexural strength of the mortars considerably diminishes (Fig. 17b–Table 7). The flexural strength of RFCP_C_0 mortar is 7 % lower, RFCP_G_0 mortar remains almost the same, and RFCP_T_0 mortar is around 10 % higher than the reference mortars OPC and OPC_LL. The presence and type of MPCM in RFCP_C and RFCP_G do not significantly affect the flexural strength of mortars. Mortars with RFCP_T sourced from ULFC with MPCM exhibit slightly lower flexural strength than mortars with RFCP_T sourced from ULFC without MPCM.

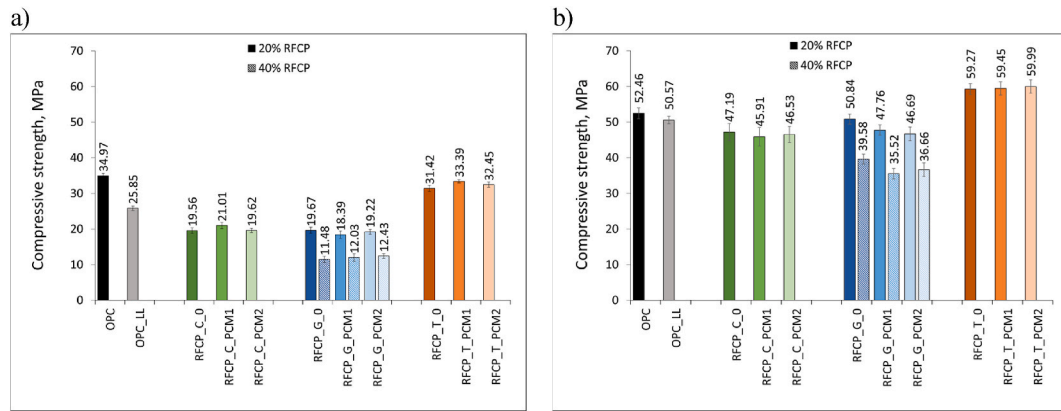


Fig. 18. Influence of RFCP type and content on compressive strength of mortars: a) after 2 days, b) after 28 days.

Table 7

The development of flexural and compressive strength of RFCP mortars related to values determined for OPC mortar, %.

Mortar	RFCP content,%	Flexural strength, % OPC mortar		Compressive strength, % OPC mortar	
		2 days	28 days	2 days	28 days
OPC	-	100	100	100	100
OPC_LL	20 (LL)	85	97	74	96
RFCP_C_0	20	70	93	56	90
RFCP_C_PCM1	20	72	92	60	88
RFCP_C_PCM2	20	64	91	56	89
RFCP_G_0	20	70	99	56	97
RFCP_G_PCM1	20	67	95	53	91
RFCP_G_PCM2	20	72	98	55	90
RFCP_T_0	20	90	105	90	113
RFCP_T_PCM1	20	87	97	95	113
RFCP_T_PCM2	20	91	101	93	114
RFCP_G_0	40	45	87	33	75
RFCP_G_PCM1	40	47	83	34	68
RFCP_G_PCM2	40	41	85	36	70

Increasing the amount of RFCP_G in the mortar from 20 % to 40 % results in approximately 35 % and 12 % further reductions in flexural strength after 2 and 28 days, respectively. The presence of MPCM in RFCP and its type do not have a significant impact on the flexural strength of mortars with 40 % RFCP_G.

The addition of 20 % RFCP in the cement significantly impacts the compressive strength of mortars after 2 days (Fig. 18a–Table 7). Mortars with RFCP_C and RFCP_G exhibit a lower compressive strength compared to the reference mortars OPC and OPC_LL by 44 % and 24 %, respectively. The compressive strength of mortars with RFCP_C aligns with that of mortars with RFCP_G, and the influence of the presence and type of MPCM in these RFCPs on compressive strength is insignificant. Mortars with RFCP_T, however, showcase a 60 % higher compressive strength than mortars with RFCP_C and RFCP_G, 30 % higher than mortars with OPC_LL, and approximately 10 % lower than OPC mortar. The source of RFCP_T (foam concrete with or without MPCM) does not affect the 2-day compressive strength.

After 28 days, the impact of RFCP on the compressive strength of the mortars diminishes but remains significant (Fig. 18b–Table 7). The addition of RFCP_C_0 and RFCP_G_0 (without MPCM) reduces the 28-day compressive strength compared to the reference mortars OPC and OPC_LL by 10 % and 7 %, respectively. Notably, the negative effect of these RFCPs on compressive strength is noticeably less than after 2 days. RFCP_G_0 mortar has slightly higher compressive strength than RFCP_C_0 mortar, although the difference is not significant. The presence of MPCM in RFCP results in a strength reduction of approximately 2 % concerning mortars with RFCP_C_0 and approximately 6 % concerning mortars with RFCP_G. Consequently, the presence of MPCM in RFCP_C and RFCP_G adversely affects compressive strength; however, the effect is not significant. The MPCM type in RFCP has a greater impact on mortar compressive strength (Fig. 19, Table 7). RFCP_T mortars exhibit significantly higher compressive strength, approximately 15 %, after 28 days than REF_OPC mortars. The origin of RFCP_T (from ULFC with or without MPCM) does not affect the compressive strength of the mortars.

Comparing the rate of compressive strength increase among the tested mortars provides interesting insights. As depicted in Fig. 19, mortars with RFCP_C and RFCP_G exhibit a lower rate of early strength increase ($R_{c2}/R_{c28} = 0.45$ and 0.40 , respectively) than OPC ($R_{c2}/R_{c28} = 0.67$) and OPC_LL ($R_{c2}/R_{c28} = 0.51$). In contrast, mortars with RFCP_T ($R_{c2}/R_{c28} = 0.55$) demonstrate a faster rate of early strength increase. The presence of MPCM in RFCP may have an insignificant impact on the rate of early strength increase, but the effect is not conclusively evident. The modest dynamics of early strength increase in mortars with RFCPs (and consequently in cement with

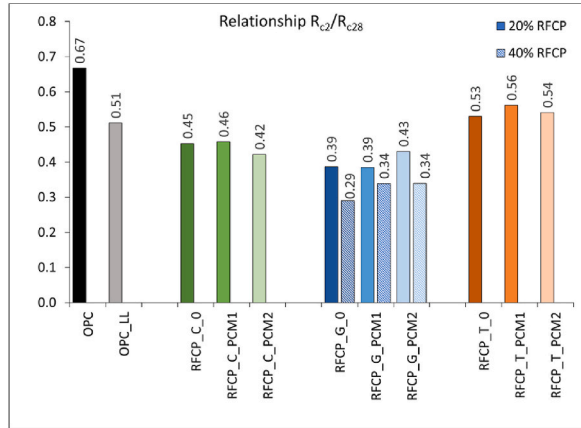


Fig. 19. Effect of RFCP on the ratio of early compressive strength development (R_{c2}/R_{c28}) in mortars.

RFCEPs) holds technological significance, influencing the timing of formwork removal.

The relation between flexural and compressive strength in the tested mortars is depicted in Fig. 20. Examining this relationship reveals that after 2 days, the ratio of flexural to compressive strength (R_f/R_c) for mortars with RFCP_C and RFCP_G is higher than that of the OPC reference mortars, and comparable to OPC_LL mortars. This can be attributed to the slower increase in compressive strength compared to flexural strength in RFCP_C and RFCP_G mortars. In contrast, for mortars with RFCP_T, the R_f/R_c ratio is similar to OPC mortars. After 28 days, the R_f/R_c ratio for mortars with RFCP_C and RFCP_G is insignificantly higher than that for OPC and OPC_LL mortars. The presence of MPCM in RFCP_C decreases the R_f/R_c ratio, while in RFCP_G, it increases, but the effect is not significant (the range of change in R_f/R_c is approximately 10 %). For RFCP_T mortars, the R_f/R_c ratio is approximately 15 % lower than OPC and OPC_LL mortars. Despite having similar flexural strengths to REF_OPC mortars, mortars with RFCP_T exhibit higher compressive strengths than the reference mortars.

The impact of RFCP on mortar strength arises from the convergence of several mechanisms. RFCP_C and RFCP_G exhibit negligible chemical activity and significantly higher water demand than cement and limestone powder (LL). The lack of chemical activity in RFCEPs evidently contributes to their negative influence on strength, particularly in the early stages. Owing to their high water demand, RFCEPs physically absorb a portion of the water (within the pores and on the surface), thereby reducing the amount of free water and subsequently lowering the water-to-binder effective (w/b_{eff}) ratio. Consequently, the adverse effect of RFCP_C and RFCP_G on 28-day strength is less pronounced than anticipated based on the amount of RFCP introduced. The grain size of RFCP_C and, despite grinding, RFCP_G is not finer than that of cement, limiting the role of RFCP as a micro filler (unlike limestone powder LL, which is finer than cement). The presence of finer RFCP_G may contribute to cement agglomeration, obstruct water access to the cement grains, disrupt hydration, and weaken the contact zone. This may explain the marginal impact of RFCP processing by grinding on mortar strength.

It's worth noting that the addition of a substantial amount of RFCP degrades the workability of mortars, potentially impeding proper compaction and resulting in a strength reduction. This effect may explain the higher-than-expected decrease in strength observed in mortars with 40 % RFCP_G. The results from hydration heat and setting time tests, as presented in Sections 3.2 and 3.3, indicate that the presence of MPCM in RFCP is expected to significantly influence mortar strength during the early hardening period. After two days, the effect of MPCM presence in RFCP_C or RFCP_G on the hydration heat of cements is negligible. This aligns with the strength test results—after 2 and 28 days, the presence and type of MPCM in RFCP have an insignificant influence on mortar strength.

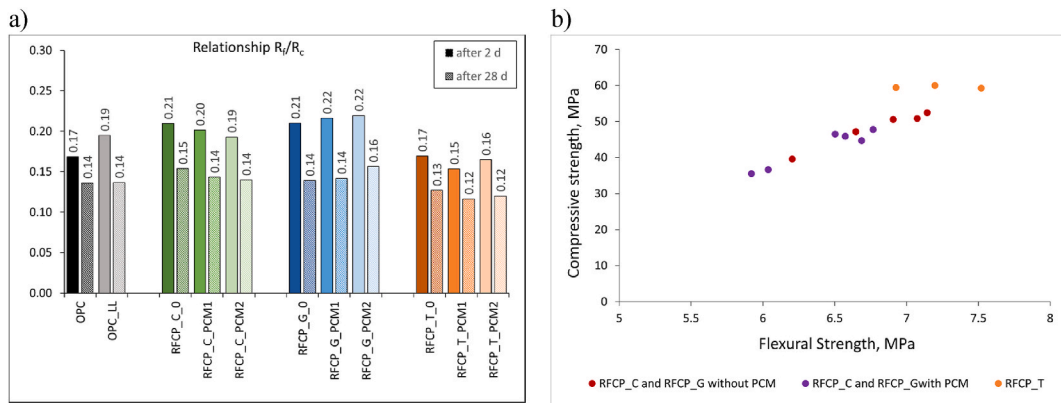


Fig. 20. Influence of RFCP on the relationship between flexural and compressive strength (R_f/R_c): a) all tested mortars, b) the impact of the RFCP type.

This effect of MPCM is expected, given the relatively small amount of MPCM. Heat-processed RFCP_T is characterized by chemical activity and high water demand. The combination of chemical activity and a reduction in w/b_{eff} results in an increase in mortar strength after 28 days compared to reference mortars.

3.8. Shrinkage of mortars

The influence of the addition of 20 or 40 % RFCP on the shrinkage of mortars is shown in Figs. 21–22. It should be noted that the registered strains refer to the total effect of autogenous and drying shrinkage measured in mortars with unprotected surface.

The shrinkage of mortars containing 20 % RFCP_C_0 or RFCP_G_0 (without MPCM) is higher after 2 and 7 days compared to OPC mortars, by 40 % and 20 % after 2 days, and about 10 % after 7 days, respectively. The influence of RFCP_C_0 and RFCP_G_0 on mortar shrinkage diminishes over time, and after 28 days, the shrinkage becomes analogous to that of OPC mortars. It's noteworthy that the effects of RFCP and limestone powder (LL) on mortar shrinkage differ, with the presence of LL clearly reducing mortar shrinkage from the outset. Increasing the addition to 40 % RFCP_G_0 causes an increase in early-age shrinkage of the mortars. However, after 14 days, the amount of RFCP_G_0 in the mortar no longer affects shrinkage. In the case of mortar with RFCP_T_0 obtained from ULFC without PCM, its shrinkage surpasses even that of corresponding mortars with RFCP_C_0 and RFCP_G_0 from day 3 onwards. After 28 days, the shrinkage of RFCP_T_0 mortars is higher than mortars with RFCP_C_0 and RFCP_G_0 by 21 % and 12 %, respectively, and higher than that of OPC by 17 %. This can be attributed to the reactivity of RFCP_T, as the hydration of the clinker phases in RFCP_T induces volume changes similar to those in clinker, while less reactive RFCP_C and RFCP_G do not exhibit the same range of volume changes.

The impact of utilizing RFCP from ULFC with MPCM differs between RFCP_T and RFCP_C and RFCP_G. The shrinkage of mortars with RFCP_T_PCM1 and RFCP_T_PCM2 is not significantly different from that of mortars with RFCP_T_0, indicating that the source of heat-treated RFCP_T (ULFC with and without MPCM) does not influence mortar shrinkage. However, the presence of MPCM in RFCP_C or RFCP_G noticeably increases the shrinkage of mortars, particularly in the case of mortars with RFCP_G.

The impact of RFCP_C and RFCP_G with MPCM on mortar shrinkage varies. In the case of mortars with RFCP_C, the presence of MPCM initially slightly reduces shrinkage. After 2 days, the shrinkage of mortars with RFCP_C_PCM1 and RFCP_C_PCM2 is lower than that of mortars with RFCP_C_0 by 6 % and 15 %, respectively. However, over time, the shrinkage of mortars with RFCP_C and PCM accelerates, surpassing that of mortars with RFCP_C_0 after only 7 days. After 28 days, the shrinkage of mortars with RFCP_C_PCM1 and RFCP_C_PCM2 is higher than that of mortars with RFCP_C_0 by 25 % and 14 %, respectively.

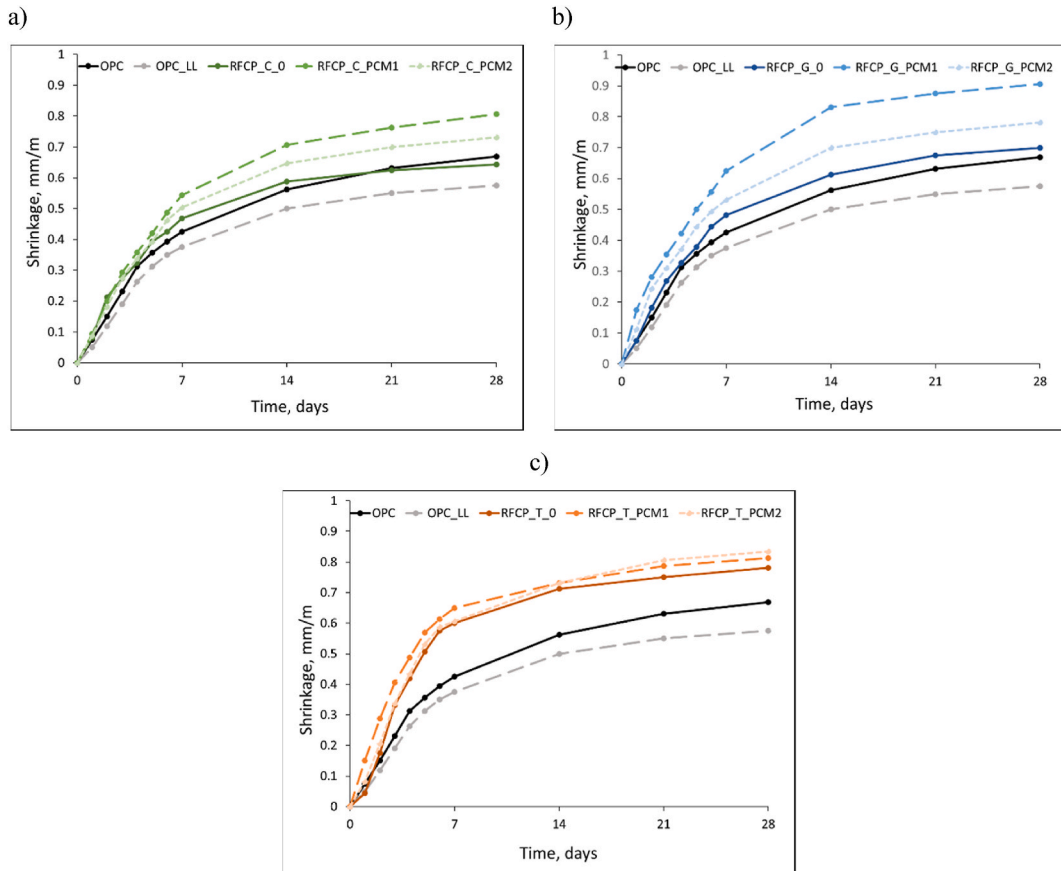


Fig. 21. Effect of 20 % RFCP on mortar shrinkage: a) RFCP_C, b) RFCP_G, c) RFCP_T.

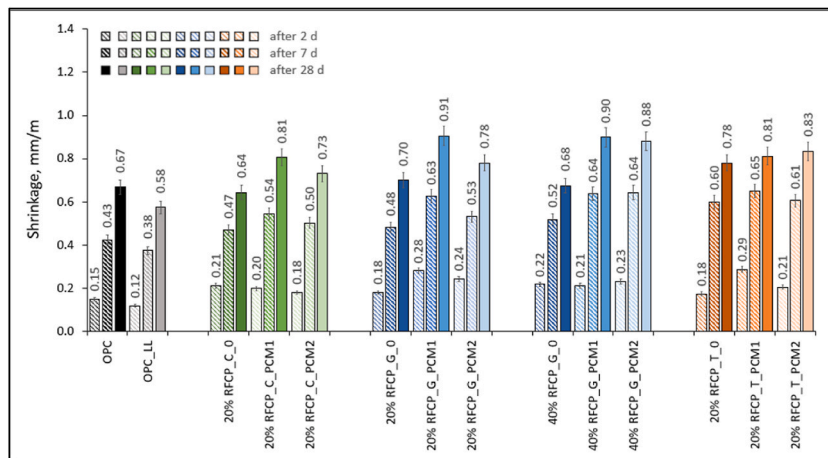


Fig. 22. Effect of RFCP type on mortar shrinkage after 2, 7 and 28 days of measurement.

For mortars with RFCP_G, the presence of MPCM significantly increases shrinkage from the outset. After 2 days, the shrinkage of mortars with RFCP_G_PCM1 and RFCP_G_PCM2 exceeds that of mortars with RFCP_G_0 by 55 % and 34 %, respectively. However, the effect of RFCP_G with MPCM on mortar shrinkage diminishes over time. After 28 days, the shrinkage of RFCP_G_PCM1 and RFCP_G_PCM2 mortars is higher than that of RFCP_G_0 by 29 % and 11 %, respectively. It's evident that the type of MPCM in RFCP also has a significant effect on mortar shrinkage, with PCM1 contributing to higher shrinkage in both RFCP_C and RFCP_G mortars.

The shrinkage of mortars with RFCP_G_PCM1 is independent of the amount of RFCP, while the shrinkage of mortars with RFCP_G_PCM2 increases with the amount of RFCP in the mix. Consequently, the type of MPCM does not affect the shrinkage of mortars with 40 % RFCP_G.

The higher initial shrinkage of mortars with RFCP without MPCM compared to OPC mortars may be attributed to its lack of chemical activity. The greater amount of chemically unbound water in mortars with RFCP may evaporate during the initial period, contributing to the higher drying shrinkage. Contrary to literature data suggesting a significant effect of RFCP on increasing mortar shrinkage after 28 days, this study did not confirm such an impact. The substantial effect of MPCM presence in the mortar on increasing shrinkage also deviates from literature data, which generally indicate a minor effect of MPCM on the long-term shrinkage of cementitious composites. The pronounced effect of MPCM type on mortar shrinkage is surprising, especially in light of its relatively minor effect on other properties of hardened mortars. These issues necessitate further research.

3.9. Water absorption of hardened mortars

The influence of RFCP type and content on the water absorption of hardened mortars is illustrated in Fig. 23. The addition of 20 % RFCP_C_0 or RFCP_G_0 increases the water absorption of mortars, reaching approximately 1 % higher than OPC mortar and 0.5 % higher than OPC_LL mortar. Thus, the grinding process of RFCP does not significantly alter the water absorption of the mortars. The presence of PCM in RFCP_C or RFCP_G leads to marginal differences in water absorption, around 0.2 %, rendering the effect of PCM presence in RFCP negligible. Increasing the amount of RFCP_G to 40 % substantially raises the water absorption of the mortars by approximately 2.5 %. Thermal processing of RFCP results in the water absorption of RFCP_T_0 mortars being slightly, around 0.2–0.5

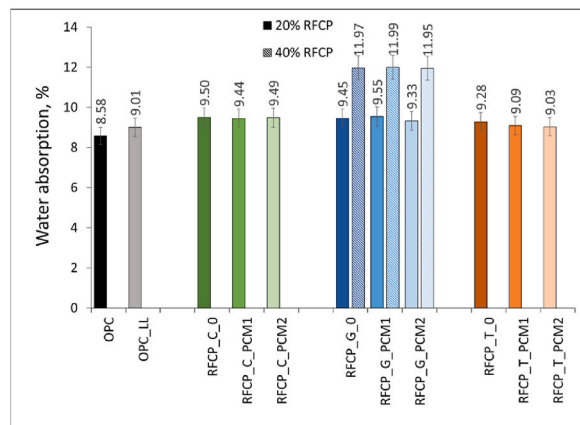


Fig. 23. Influence of RFCP type and content on water absorption of hardened mortars.

%, lower than that of RFCP_C_0 and RFCP_G_0 mortars but higher than OPC mortar. The source of thermally processed RFCP_T (from ULFC without or with PCM) does not influence the water absorption of mortars.

The impact of RFCP on water absorption in mortars arises from the convergence of several mechanisms. The negligible (RFCP_C and RFCP_G) or low (RFCP_T) chemical activity of RFCP, introduced as a portion of cement, contributes to a more porous paste structure. Simultaneously, the high water demand of RFCP reduces the water-to-binder effective (w/b_{eff}) ratio, contributing to the densification of the paste and mortar structure. While the addition of 20 % RFCP negatively affects the workability of fresh mortars, it is not significant enough to hinder proper compaction. Consequently, the effect of adding 20 % RFCP on the water absorption of mortars is adverse but not overly pronounced. The substantial increase in water absorption in mortars with 40 % RFCP_G is due to the significant reduction in cement caused by RFCP's high water demand. This reduction in w/b_{eff} ratio, coupled with a substantial deterioration in the workability of mortars, makes proper compaction challenging.

3.10. Thermal conductivity and volume heat capacity of hardened mortars

The influence of RFCP type and content on the thermal conductivity of mortars is depicted in Fig. 24a. Mortars with 20 % RFCP_C_0, RFCP_G_0, or RFCP_T_0 exhibit thermal conductivities lower than OPC mortar by 12 %, 10 %, and 3 %, respectively. The presence of MPCM in RFCP_C and RFCP_G reduces the thermal conductivity of RFCP mortars by approximately 2 %, with RFCP_C_PCM2 showing a higher reduction at 10 %. Increasing the amount of RFCP_G to 40 % results in a 26 % reduction in the thermal conductivity of mortars compared to OPC mortar, and the presence of PCM further reduces thermal conductivity by 2 %. Notably, the introduction of limestone powder LL, unlike RFCP, slightly increases the thermal conductivity of mortars compared to OPC mortars.

In general, thermal conductivity can be linked to the density of mortars, being higher with increased density (Fig. 24b) [102–105]. Lower density in mortars is usually linked to an increased amount of air in pores of the matrix, and as the cement matrix and most commonly used normal weight or heavyweight aggregate are highly conductive to heat, an increased amount of air serves to decrease the conductivity [105–107]. The correlation between density and thermal conductivity has been extensively investigated, particularly in the context of lightweight concrete. Equations formulated by ACI Committee 213 R-03 [108] and several other models [105] have been proposed to estimate thermal conductivity based on density in this regard.

The introduction of RFCPs and the presence of MPCMs reduce the density of the mortar, consequently lowering the thermal conductivity of mortars with their addition. The lower thermal conductivity of RFCP_C_PCM2 mortar is not primarily attributed to MPCM presence but rather to the lower density of the mortar, possibly resulting from the lower compaction of the test sample. Based on this observation, it can be inferred that the effect of MPCM presence in RFCP on the thermal conductivity of mortars is insignificant.

The impact of RFCP type and content on the volume heat capacity of mortars is illustrated in Fig. 25. Volume heat capacity measurements were conducted at 20 °C, a temperature that does not allow for the detection of potential heat accumulation by the MPCM, given its transition temperature of 24 °C or 37 °C. The presence of RFCP has an insignificant effect on the volume heat capacity of the mortars. In comparison to OPC mortars, the volume heat capacity of mortars with RFCP, regardless of their type and the presence of MPCM, does not differ by more than 10 %. No clear trends in the influence of RFCP type, including the presence of MPCM, can be identified.

4. Conclusions

Based on the results of the study regarding the influence of recycled foam concrete powder (RFCP) derived from ultralight foam concretes (ULFC) with and without microencapsulated phase change materials (MPCM) on the properties of cement and mortar, the following conclusions can be drawn.

- Overall, substituting 20 % or 40 % of Ordinary Portland Cement (OPC) with RFCP, regardless of its origin (without or with MPCM) and the processing method, significantly impacts the properties of both cement and mortar. OPC blended with RFCP exhibits a notably higher water demand than OPC and OPC with limestone (OPC_LL), delayed initial setting time, decreased rate of heat

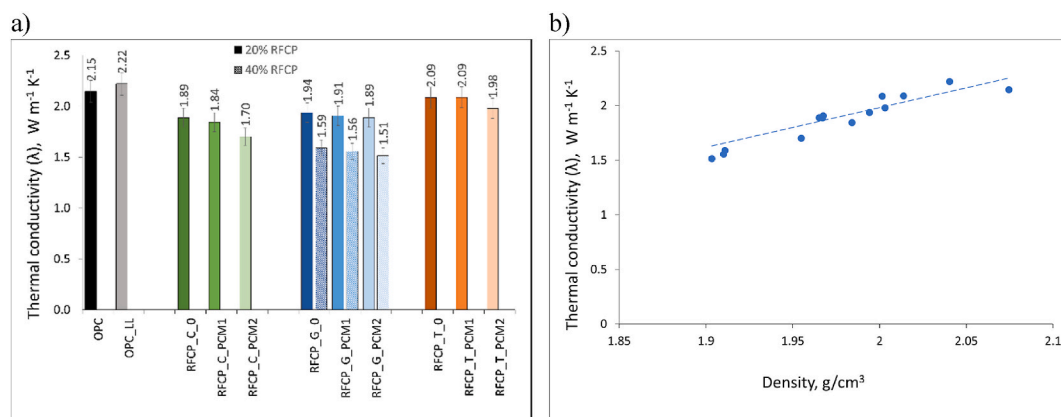


Fig. 24. a) The influence of type and content of RFCP on the thermal conductivity of mortars, b) the relationship between thermal conductivity and density of mortars.

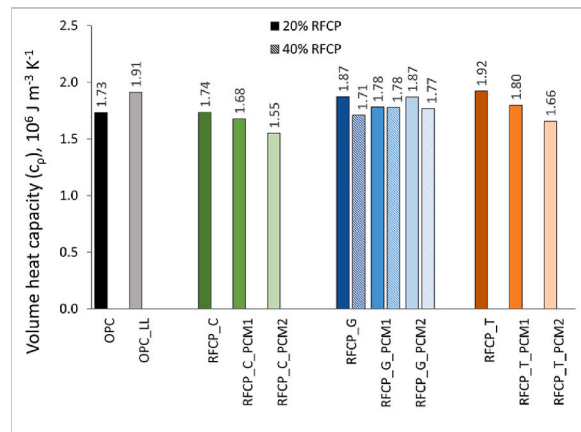


Fig. 25. The influence of type and content of RFCP on volume heat capacity of mortars.

evolution, and reduced total heat evolution. Fresh mortars containing OPC with 20 % RFCP replacement demonstrate considerably lower flowability, significantly faster loss of flowability, and slightly higher air content compared to OPC and OPC_LL mortars. Regarding hardened mortars and compared to OPC-based mortars, those with OPC blended with RFCP show a lower rate of early strength gain, lower compressive and flexural strength after 2 and 28 days (except for OPC with RFCP after heat treatment), increased shrinkage, and higher water absorption. The presence of RFCP in cement reduces mortars' thermal conductivity and affects volume heat capacity without displaying clear trends.

- The presence of MPCM in the RFCP enhances the above-mentioned effects if the simple processing method as crushing is used. Simultaneously, utilizing processing methods such as grinding, or heat treatment substantially mitigate or even counteract the negative effects of MPCM presence in RFCP on the properties of cement and mortar. Generally, the type of MPCM in the RFCP does not influence the most properties of the cement and mortar.
- The processing method of RFCP significantly influences the properties of cement and mortar when RFCP is used as a partial replacement for cement. Compared to OPC, OPC with RFCP crushed and sieved to a grain size $<0.125 \text{ mm}$ (or $\text{SSA} \sim 6000 \text{ cm}^2/\text{g}$) demonstrated higher water demand, delayed cement setting time, and reduced hydration heat during initial cement hydration. Mortars with RFCP after crushing processing exhibit impaired flowability, lower strengths, increased water absorption, higher shrinkage, lower thermal conductivity and volume heat capacity compared to OPC mortars. Grinding and sieving RFCP to a grain size $<0.063 \text{ mm}$ (or $\text{SSA} 6000\text{--}7500 \text{ cm}^2/\text{g}$) reduces water demand compared to using RFCP only crushed. The negative impact of RFCP after grinding on mortar workability is significantly less than crushed RFCP, enabling its use in larger quantities. Generally, the impact of grinding compared to crushing of RFCP on the discussed properties is positive since mitigating the undesirable deterioration of cement and mortar properties. Heat treatment of RFCP increases water demand compared to RFCP subjected to crushing and grinding and as a result significantly impairs mortar flowability. After heat treatment, employing RFCP as a partial substitute for OPC delays the setting time and diminishes the rate of heat evolution, though to a lesser degree than RFCP that has been crushed or ground. Mortars incorporating heat-treated RFCP demonstrate notably greater strengths than those utilizing RFCP post-crushing and grinding, albeit with higher levels of shrinkage.
- Finally, all mortars containing RFCP as a 20 % substitution rate of OPC, regardless of the presence of MPCM, exhibited compressive strengths surpassing 16 MPa after 2 days and 42.5 MPa after 28 days. This meets the requirements of EN-196-1 for cement classified as 42.5, indicating the feasibility of producing CEM II/A-F 42.5 cement using RFCP derived from foam concrete also containing MPCM. Despite its generally negative impact on compressive strength, this underscores the potential utility of such RFCP. However, it should be remembered that the presence of MPCM prolongs setting time and increases the water demand for cement, posing challenges to practical usage. To mitigate these issues, RFCP with MPCM should at least undergo grinding before use, reducing its adverse effects on cement water demand and setting time, ultimately achieving acceptable technological properties. While thermal treatment allows MPCM to be extracted from RFCP and enhances the strength of hardened mortar, it does not seem to be the optimal solution. Thermally processed RFCP has a very high water demand, posing technological challenges for its use in cement or as an additive to mortars.

CRedit authorship contribution statement

Barbara Klemczak: Writing – review & editing, Writing – original draft, Validation, Supervision. **Jacek Gołaszewski:** Methodology, Investigation, Supervision, Writing – original draft, Writing – review & editing. **Grzegorz Cygan:** Visualization, Methodology, Data curation, Investigation. **Małgorzata Gołaszewska:** Writing – review & editing, Writing – original draft, Visualization, Methodology, Investigation, Data curation. **Henk Jonkers:** Writing – review & editing, Supervision, Conceptualization. **Dmitry Zhilyaev:** Writing – review & editing, Supervision, Conceptualization. **Eduardus A.B. Koenders:** Writing – review & editing, Supervision, Conceptualization.

Declaration of competing interest

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

Data availability

Data will be made available on request.

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