

Article

Green Manufacturing of UHPFRC Made with Waste Derived from Scrap Tires and Oil Refineries

Hassan Abdolpour ¹, Murugan Muthu ¹, Paweł Niewiadomski ¹, Łukasz Sadowski ^{1,*}, Łukasz Hojdys ², Piotr Krajewski ² and Arkadiusz Kwiecień ²

¹ Department of Materials Engineering, Wrocław University of Science and Technology, 50-372 Wrocław, Poland; hassan.abdolpour@pwr.edu.pl (H.A.); murugan.muthu@pwr.edu.pl (M.M.); pawel.niewiadomski@pwr.edu.pl (P.N.)

² Faculty of Civil Engineering, Cracow University of Technology, 31-155 Cracow, Poland; lukasz.hojdys@pk.edu.pl (L.H.); piotr.krajewski@pk.edu.pl (P.K.); arkadiusz.kwiecien@pk.edu.pl (A.K.)

* Correspondence: lukasz.sadowski@pwr.edu.pl; Tel.: +48-713-203-742

Abstract: Ultrahigh-performance fiber-reinforced cement-based composite (UHPFRC) made with waste derived from scrap tires and oil refineries was tested in this study. The UHPFRC sample exhibited a maximum compressive strength of about 189 MPa at the end of 28 days. Steel fibers were recovered from scrap tires and were added up to 3% by volume in the UHPFRC samples. Such additions reduced cement flow by 11% but improved compressive strength by 21%. The equilibrium catalyst particles (ECAT) disposed of by oil refineries were used in amounts of up to 15% by weight as a replacement for cement in such UHPFRC samples. These aluminosilicate materials are spherical in shape and have a porous microstructure, which was found to reduce the cement flow by absorbing more free water onto their surfaces. They also reduced the heat and strength developments at early stages. However, the total cost of the final cement-based mixture and associated CO₂ emissions were reduced by up to 7% and 15% due to the inclusion of the ECAT particles. These findings help to optimize the ECAT and recovered steel fibers in the UHPFRC mix design, and such waste valorization strategies can help achieve the goal of becoming carbon neutral.

Keywords: equilibrium catalyst; recycled steel fiber; cement; properties; CO₂ emissions



Citation: Abdolpour, H.; Muthu, M.; Niewiadomski, P.; Sadowski, Ł.; Hojdys, Ł.; Krajewski, P.; Kwiecień, A. Green Manufacturing of UHPFRC Made with Waste Derived from Scrap Tires and Oil Refineries. *Appl. Sci.* **2024**, *14*, 5313. <https://doi.org/10.3390/app14125313>

Academic Editors: Sara Cattaneo and Manuela Alessandra Scamardo

Received: 30 April 2024

Revised: 14 June 2024

Accepted: 18 June 2024

Published: 19 June 2024



Copyright: © 2024 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (<https://creativecommons.org/licenses/by/4.0/>).

1. Introduction

Ultrahigh-performance fiber-reinforced cement-based composite (UHPFRC) exhibits excellent properties and is used more widely in long-span bridge deck constructions [1]. The application of UHPFRC reduces the thickness of the deck by up to 35%, resulting in cost savings associated with materials, installation, and labor [2]. The production of UHPFRCs requires a larger volume of cementitious materials, which improves the properties related to long-term durability by densifying the microstructure [3]. UHPFRCs must have a high packing density in order to obtain superior mechanical and durability properties [4]. Limestone, silica fume, fly ash, slag, and quartz powder, as replacements for cement, improve the packing density. Discrete steel fibers are recommended in the design of UHPFRC to effectively inhibit crack propagation under vehicle loads [5]. Today, the consumption of energy and natural resources is becoming restricted, and the construction sector around the world is paying more attention to the use of waste utilization strategies in concrete production [6]. Despite its excellent properties, UHPFRC is not considered favorably due to the higher cost of ingredients and the fact that its production has a high carbon footprint [7]. The overuse of cement and steel fiber affects the production cost of UHPFRC, and also causes CO₂ emissions [8]. This implies that UHPFRC needs to be further optimized with low-carbon materials, with its final cost and ecological developments requiring more experimental information, which is the main aspect in this research. The use of industrial by-products in UHPFRC reduces the environmental and economic impacts [9].

However, their quantities are restricted because they exhibit weak strength gain at early stages. The proposal of using low-cost material with greater sustainability remains a challenge. A major concern for the diversity of environmental agencies is to acquire a productive methodology to manage waste products. Since a lot of solid waste goes to landfills in sub-urban and rural areas, this can also cause, in addition to environmental pollution by occupying the landscape, some health problems [10].

Unlike the end-of-life phase of concrete [9], of which considerable studies have been conducted, little attention has been paid to the waste disposed of by the petrochemical industries. In crude oil refinery factories, one of the indispensable materials (in a powder form) is fluid cracking catalyst (FCC), which is vital for breaking the molecules of high-weight crude oils. Since the cracking process requires high temperatures, the FCC is disactivated after some time and should be replaced by the new one. The spent equilibrium catalyst (ECAT) suggested for use in this study is a waste material that is generally disposed of in landfills [8]. The ECAT was tested as being characterized by pozzolanic activity. The formation of calcium silicate hydrate (CSH) gel makes ECAT more beneficial for the production of concrete mixes, which is due to the fact that it reacts with the $\text{Ca}(\text{OH})_2$ produced during the hydration of cement [11]. The benefits of using ECAT in the production of cementitious products such as paste [12], mortar [13], concrete [14], and UHPFRC [15] are presented in the literature. ECAT particles have a porous structure, and coarser particles were used as a replacement for sand in paper [15], with fine powders being used as a supplementary cementitious material in paper [12]. Coarser particles absorb free water, which acts as an internal curing agent in concrete mixes [15]. In these cases, there is an increase in mechanical properties and a decrease in the workability of ECAT-based concrete mixes. The increase in mechanical properties can be justified by the fact that the reaction of ECAT with calcium hydroxide (CH) could result in the production of CSH gel, calcium aluminate hydrate (CAH), and calcium aluminosilicate hydrate (CASH), which are fundamental in improving the mechanical performance of cement-based mixes [16]. Regarding the decrease in workability, the main justification for this fact is that ECAT, because of its larger surface area and highly porous microstructure, increases the potential for a high capacity of water absorption.

The reinforcement of concrete matrices using discontinuous fibers is a widely recognized technology for improving mechanical properties, including the postcracking behavior of materials [17]. After the onset of a crack, the fibers exert their exertions to arrest the crack using a bridging action and in turn improve the post-peak and residual strength of the structural elements [18]. The initial high cost of UHPFRC is due to the use of steel fibers. In addition to material costs, sustainability problems in steel production should be taken into account [19]. Each ton of steel is estimated to emit 1.9 tons of CO_2 [20]. Therefore, the use of steel fibers recovered from scrap tires could be beneficial in terms of reducing the initial cost and increasing the sustainability of UHPFRC mixes. The performance of UHPFRC containing different amounts of this recovered steel fiber (RSF) has been studied in the literature [8]. The increase in the amount of RSF has been shown to adversely affect not only the workability but also the homogeneity of the fresh mixture [21]. During mixing, the fibers will tend to agglomerate, and this balling effect reduces the workability of fresh mixes [22]. Some recommendations have been proposed to avoid this balling effect, such as the addition of incremental fiber [23], scattering after wet mixing [24], and using a fiber aspect ratio of 200 [25]. The importance of rubber being attached to RSF with regards to concrete performance has been highlighted. A decrease in compressive strength was reported from 136 MPa to 130 MPa in the case of RSF with rubber attached, while a decrease of 5% in compressive strength was reported at 141 MPa in the case of fibers without rubber attached [23]. In the case of the flexural toughness of UHPFRC containing RSF, a gradual increase in post-peak behavior was reported by increasing the percentage of fiber [26]. The beneficial use of RSF has also been reported to provide crack bridge and crack-arresting actions to improve the post-peak behavior of tested beams under flexural load [27]. A relatively small amount of residual flexural strength, as well as softening responses, was

revealed with a lower content of fiber use [28], with a significant improvement in flexural strength being observed when the amount of steel fiber was increased [29]. Improvements in the first crack by 60% and maximum load by 20% were reported when using 60 kg/m³ of RSF [30]. Recently, the mechanical performance of affordable and eco-efficient UHPFRC containing RSF was studied [31]. In addition to studying short fiber and the effect of clarity, different UHPFRCs containing 2%, 3%, and 4% of fiber were proposed. Fibers less than 9 mm were found to significantly reduce the mechanical strength of the proposed mixtures, and the clarity of the fiber was also shown to improve performance [32]. Furthermore, a significant cost and environmental benefit of using RSF in the production of UHPFRC was highlighted in paper [33].

An economic solution for recycling ECAT and RSF would be their utilization in the production of concrete mixes for infrastructure applications. The synergistic effects of the RSF and ECAT dose on the optimization, performance, and environmental impacts of the UHPFRC have not yet been studied. To fill this research gap, UHPFRC samples containing different amounts of ECAT and RSF were tested in this study. Their rheological, mechanical, and microstructural properties were evaluated using flow table apparatus, a universal testing machine, and a scanning electron microscope. The economic and environmental impacts of the developed UHPFRC were also highlighted with regards to cement-based mixes that are pertinent to the Polish construction industry, together with a discussion of the different limits of the concrete system and the conversion factors involved. These findings can allow for the development of sustainable UHPFRCs in the European market, where the availability of RSF and ECAT is abundant and the demand for bridge deck construction is high.

2. Materials and Methods

2.1. Sample Preparation

Portland cement of grade CEM I 52.5R that complies with EN 197-1 [34] was used to prepare the different samples in this study. Other raw materials were river sand, silica fume, fly ash, spent equilibrium catalyst, potable water, liquid admixture, and recycled steel fibers. In this study, river sand particles of sizes less than 5 mm were used. The Sika ViscoCrete-93 model superplasticizer, based on sulfonated naphthalene formaldehyde (SNF), was used to improve cement flow. The Orlen petrochemical company provided the ECAT in powder form, while steel fibers mechanically extracted from scrap tires were supplied by a local source. The average specific gravity and water absorption of the aggregates were calculated to be 2.67 and 0.43%. These characteristics were determined according to EN 1097-6 [35].

The cementitious materials were loaded onto a circular metal disc and scanned with Rigaku model X-ray fluorescence (XRF) in order to obtain information regarding their oxide composition. Table 1 lists the XRF results. The loss in ignition of these materials was found by heating them up to 1000 °C in a hot air oven. The mean values were found to be very low, indicating that the materials were free of organic matter and carbon species. The ECAT particles were derived from zeolite, as their main oxides were found to be SiO₂ and Al₂O₃. The SEM image in Figure 1 shows the spherical morphology of the ECAT particles. The sample was mounted on a steel stub using a carbon adhesive, then gold-coated for 60 s, and scanned in a JEOL JSM-6610A model SEM at higher voltage to obtain morphological information. Figure 1 shows the particle size distribution of the cementitious materials, which was obtained using the laser diffraction technique. The Cilas 1090 particle size analyzer was used in this study. The mean particle size of the cement, fly ash, and ECAT was determined to be 146, 128, and 251 µm, respectively. The average size of the silica fume reported by the manufacturer was 0.27 µm. A vernier caliper was used to measure the length and diameter of the RSF samples. Their diameter was found to fall within 0.06–1.6 mm, while their length was 5–35 mm. The cement-based mixtures listed in Table 2 were designated based on the variations in the amount of RSF and ECAT, and they were proportioned according to the Andreasen and Andersen particle packing model adopted in Yu, et al. [36]. These mixes were proportioned with a low water-to-binder (w/b) ratio

of 0.16. The sand-to-binder ratio was maintained at 0.92 by weight in all the mixes. A superplasticizer was added up to 2.2% by weight of the cementitious material in order to improve workability. The raw materials were mixed in a high-speed Hobart mixer in a laboratory environment for up to 8 min, and the fresh mixture was filled into iron molds of different sizes and consolidated using a vibration table. The upper surfaces were leveled with a flat blade, and the fresh samples were demolded after 24 h and allowed to cure in tap water for up to 28 days. The temperature and relative humidity of the curing environment was maintained at 25 °C and 65%.

Table 1. XRF results of the raw materials.

Sample	CaO	SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	SO ₃	MgO	Na ₂ O	K ₂ O	Ignition Loss
Cement (%)	64.6	21.4	3.7	4.6	1.5	0.8	0.3	0.4	2.7
Silica fume (%)	0.1	94.2	0.8	0.3	0.4	0.5	0.3	1	2.4
Fly ash (%)	5.2	46.8	26.2	9.9	-	2.7	2.8	5.5	0.9
ECAT (%)	-	54.2	38.1	-	3.9	3.4	-	-	0.4

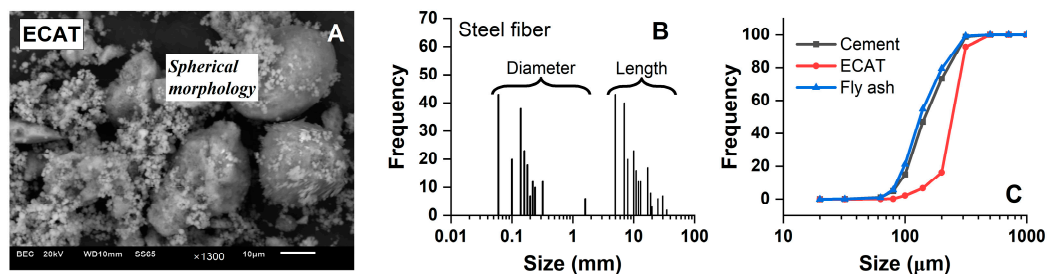


Figure 1. (A) SEM image of the ECAT, (B) dimensions of the RSF, and (C) PSD of the cementitious materials.

Table 2. Mix details of the different cement-based samples.

Sample Designation	Steel Fiber (% by Volume)	Cementitious Materials (% by Weight)			
		ECAT	Cement	Fly Ash	Silica Fume
S0E0	0	0	71	13	16
S0E5	0	5	66	13	16
S0E10	0	10	61	13	16
S0E15	0	15	56	13	16
S1E0	1	0	71	13	16
S1E5	1	5	66	13	16
S1E10	1	10	61	13	16
S1E15	1	15	56	13	16
S2E0	2	0	71	13	16
S2E5	2	5	66	13	16
S2E10	2	10	61	13	16
S2E15	2	15	56	13	16
S3E0	3	0	71	13	16
S3E5	3	5	66	13	16
S3E10	3	10	61	13	16
S3E15	3	15	56	13	16

2.2. Test Methods

A Calmetrix I-Cal 8000 HPC was used to monitor the heat developments for six days at 30 °C and 65% relative humidity (RH) in laboratory circumstances, in accordance with ASTM C1702 [37]. Approximately 30 g of fresh cement paste was put into the calorimeter channel within a few seconds of mixing. Four paste samples containing ECAT at concentrations of 0, 5, 10, and 15% were prepared using a water-to-binder ratio of 0.4. The flow table

apparatus recommended in ASTM C230 [38] was used to assess the workability of the fresh cement mixes. The fresh sample was loaded into the firmly placed truncated cone at the center of the table. The cone was then removed, leaving the sample, which was dropped 15 times. The flow diameter was measured using a standard ruler, and the average of three replicates was calculated. A mortar sample of $40 \times 40 \times 40 \text{ mm}^3$ was destroyed on a Zwick Roell universal testing machine at a rate of 0.6 MPa/s according to EN 12390-3 [39]. The failure load was divided by the cross-sectional area to calculate the compressive strength. The average of four replicates were calculated.

In accordance with ASTM C348 [40], a mortar sample measuring $40 \times 40 \times 160 \text{ mm}^3$ was utilized to ascertain the flexural strength. Both notched and unnotched samples were tested. Using a precision saw with a diamond tip, a rectangular notch measuring 2 mm in thickness and 10 mm in depth was cut. The Zwick 1455 UTM was employed, which was outfitted with a QuantumX model data-gathering system. To record load variations, a load cell with a capacity of 20 kN was employed. Using a stiff semi-cylinder with a constant displacement of 0.5 mm per min, the load was released at the center of the sample. A rigid steel profile held the entire system in place to prevent sample distortion during loading, and two rigid semi-cylinders set 120 mm apart supported the samples. After that, an HBM WA10 linear variable displacement transformer (LVDT) with a 10 mm stroke was used to instrument the samples. To measure vertical deformations, LVDT was vertically mounted on the samples. This three-point bending technique was applied to unnotched samples, and the average of three replicates was computed. Equation (1)—where P is the applied load, l is the length between the roller supports, and b and h are the sample's width and height—was used to determine the flexural strength in the sample's center.

$$\text{Flexural strength (MPa)} = \frac{3Pl}{2bh^2} \quad (1)$$

2.3. Scanning Electron Microscopy

The UHPFRC samples were cut into multiple pieces using a diamond-tipped precision saw. They were dried, epoxy-impregnated using a Buehler model vacuum impregnation system, and left undisturbed for 12 h. The hardened epoxy polymer was polished on a metal disc at a speed of 300 rpm, followed by polishing again at 150 rpm using three different polycrystalline diamond sprays (of sizes 9, 3, and $1 \mu\text{m}$). The Buehler Metaserv-250 model polishing machine was used. The fine polished surfaces were then imaged using SEM at a current of 20 kV with a backscattered-electron mode. Carbon adhesive was used to mount the hardened epoxy polymer onto the metal stubs that were loaded into the microscope.

3. Results and Discussion

3.1. Heat of Hydration

Figure 2 shows the results of the isothermal calorimetry test. The cumulative heat exhibited by the fresh cement pastes modified with 0, 5, 10, and 15% of ECAT was determined to be 344, 329, 310, and 287 J/g of binder. These pastes were made using a weight w/b ratio of 0.4 by weight and exhibited an exothermic reaction when water was mixed with cement. They released the maximum amount of heat at 14 h from the time of mixing. The addition of 15% ECAT reduced the generation of cumulative heat by 17% at the end of the experiment. There was a gradual decrease in the development of heat from the cement mixes due to the increasing dose of ECAT. However, the ECAT additions did not influence the time at which the maximum amount of heat was released by the fresh mixes. These findings explain that the inclusion of ECAT reduced the degree of cement hydration but did not cause any setting delays. Silica fume [41], fly ash [42], and ECAT [43] influenced the hydration kinetics of the clinker compounds and the formed cement hydrates. Fly ash and ECAT were used at higher volumes, and their reaction was slower than that of the clinker compounds. This was reflected in the assemblage of cement components, the microstructure, and the performance of the cement matrix. They continue to react in the

long term, which contributes to the development of compressive strength during prolonged hydration [44]. da Cunha, et al. [45] used thermogravimetry to assess the effect of the size of ECAT on the degree of the pozzolanic reaction. ECAT particles with a size smaller than 37 μm are able to form a relatively higher amount of $\text{Ca}(\text{OH})_2$ in cement-based mixes at an early stage [45]. In this study, the average size of the ECAT was found to be almost two times larger than that of the fly ash, but the pozzolanic reaction of the siliceous fly ash could be relatively slow due to the higher presence of the glassy-phase content. The reactivity of low-calcium fly ash particles is associated with the rate of dissolution of the glassy phase and the release of reactive species [46].

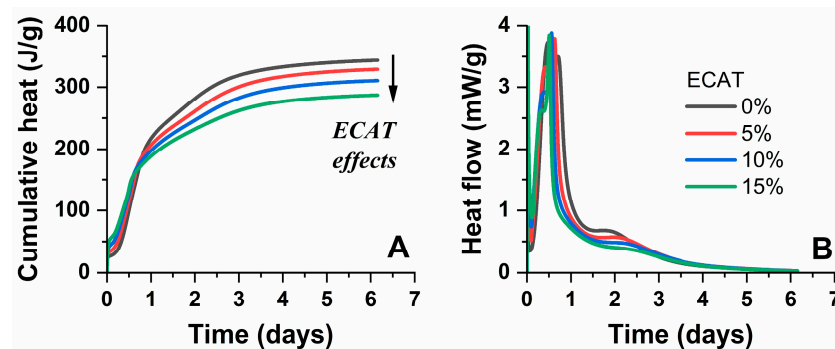


Figure 2. Results of the isothermal calorimetry test: (A) cumulative heat and (B) heat flow curves.

3.2. Performance and Microstructure

The S0E0, S0E5, S0E10, and S0E15 samples do not contain RSF. Figure 3 illustrates their fresh and hardened properties. There is a gradual decrease in the flow diameter of the fresh cement-based samples due to the increase in ECAT. The cement flow was reduced by 17% due to the use of ECAT by up to 15%. The spherical particles were mostly porous, as more free water was absorbed onto their surfaces than on the cement particles, which in turn affects the flowability of the cement-based mixes. At the end of 28 days, S0E0 was found to reach a maximum compressive strength of approximately 155 MPa. The addition of ECAT reduced this strength gain by up to 17%, which is consistent with the results of the calorimetry test. Replacement of the cement with ECAT reduced heat developments in the fresh matrices, indicating that there is a relatively lower formation of cement hydrates that give compressive strength. Due to the fact that ECAT is an aluminosilicate material derived from zeolite, it can induce a pozzolanic reaction in combination with silica fume and fly ash and also generate secondary CSH at later stages. Such reactions would also densify the microstructure, indicating the improvement in long-term durability. The average flexural strength of S0E0, S0E5, S0E10, and S0E15 was determined to be 23, 25, 28, and 25 MPa, suggesting that the addition of ECAT improves the flexural capacity of ultrahigh-performance cement-based matrices at early stages. The flexural strength improved to 22% due to the replacement of cement with ECAT up to 10%. A higher cement content was used in the preparation of these mixes, which obviously improved brittleness and limited the gain in tensile strength. This weak mechanical property can be improved with the use of ECAT instead of slag, fly ash, and silica fume. The SEM images shown in Figure 4 revealed the microstructure of different cement-based samples at the end of 28 days. The darker regions are attributed to empty pores and voids, while the grey regions were solid components. The unhydrated cement grains were relatively brighter than the fly ash, ECAT, and cement hydrates. Spherical fly ash particles and angular unhydrated cement grains were observed in the microstructure of S0E0, while the uniform dispersion of spherical ECAT particles was observed in addition to them in S0E15. The microstructure of these samples was dense, which is due to the fact that the cementitious materials were able to effectively fill the pores in the matrix. A minor color change was observed on the periphery of most fly ash and ECAT particles, indicating that chemical reactions with cement grains

could have been initiated. Cenospheres are hollow particles in fly ash and were seen in the microstructure of S0E0.

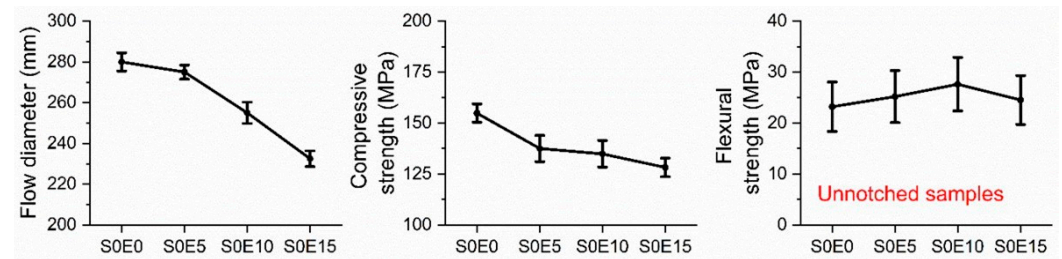


Figure 3. Various properties of the cement-based mixes containing different amounts of ECAT.

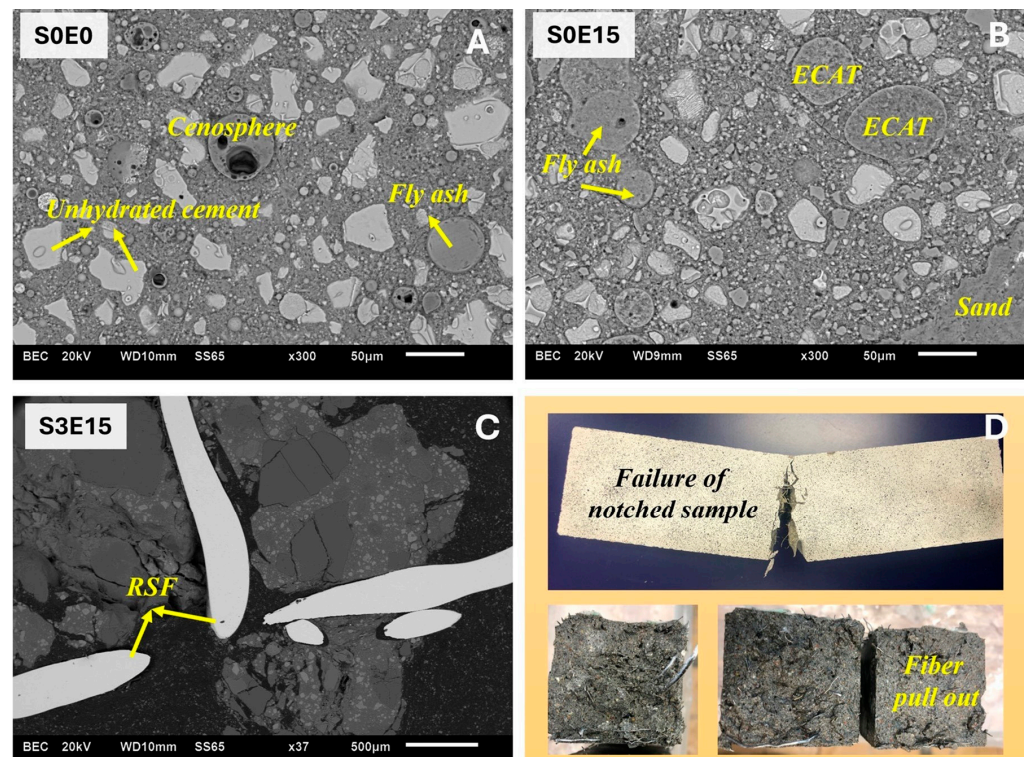


Figure 4. SEM images revealing the microstructure of (A) S0E0, (B) S0E15, and (C) S3E15, and (D) digital images revealing the fiber pull-out failure of the tested samples.

The optical image in Figure 5 revealed the cut-section of the UHPFRC sample. Scrap rubber detached from the surface of the RSF was observed in such a cut-section. The addition of RSF reduces the fluidity of the fresh cement-based mix, which is evident in this figure. Steel fibers tend to interlock, in turn affecting mix workability. Biswas, et al. [47] found that the aspect ratio and quantity of steel fibers greatly influence cement flow. Figure 6 shows the various properties of the UHPFRC samples. The mean flow diameter of S1E0, S1E15, S2E0, S2E15, S3E0, and S3E15 was calculated to be 275, 223, 265, 200, 248, and 179 mm. There is a gradual decrease in the flow diameter as a result of the increase in the dose of RSF and ECAT. Increasing the dose of RSF reduced the cement flow by 10%. However, the addition of ECAT reduced cement flow by 28% in the presence of the maximum amount of RSF. The addition of up to 1% RSF to cement-based mixes has no effect on compressive strength, which can be attributed to the combined effect of inadequate RSF dispersion and mixture compaction. The average compressive strength of S1E0, S1E15, S2E0, S2E15, S3E0, and S3E15 was determined to be 152, 130, 166, 144, 187, and 165 MPa. The compressive strength of the cement mixes was found to increase by up to 23% when the addition of a dose of steel fibers increased from 1% to 3% by volume. The addition

of ECAT up to 15% by weight reduced this strength gain by up to 12% in the maximum presence of steel fibers. The flexural property of the UHPFRC is characterized by a residual strength in the postcracking stage with a substantial enhancement in the toughness of the cement-based composite. Steel fibers in the cement-based mix prevent and control crack spread. Figure 6 shows the flexural strength of the UHPFRC samples that were notched prior to testing. There is a gradual increase in the flexural strength of the UHPFRC samples when the RSF in them increased from 1% to 3% by volume. The average flexural strength of the notched samples labeled S1E0, S1E15, S2E0, S2E15, S3E0, and S3E15 was found to be 5.7, 5.4, 6.4, 12.1, 17.6, and 15.8 MPa. Increasing the ECAT did not affect the flexural behavior of the UHPFRC containing RSF up to 2%. In contrast, there is a marginal decrease in the flexural strength of the 3% RSF-reinforced UHPFRC samples with an increasing amount of ECAT. Cement binds RSF and sand together in the UHPFRC matrix, and its replacement with pozzolanic materials affected the tensile properties only in the case of a higher volume of RSF (i.e., 3% by volume). Steel fibers tend to pull out of the binder matrix under tensile loads, and the addition of higher volumes of RSF demands more cement in the matrix to avoid weak mechanical performance. Figure 4 shown the microstructure of a UHPFRC sample where a small gap was observed at the interface between the RSF and the binder matrix, indicating that the steel fibers tend to pull out under applied loads (see Figure 4D). The prismatic UHPFRC samples were notched in the center as they were destroyed by flexure, with a major crack forming through the depth of the sample. The random distribution and orientation of steel fibers in UHPFRC samples usually complicate the stress fields of the crack tip [48], and the local tensile strength and fracture toughness can be seen to be heterogeneous [49]. The UHPFRC samples were found to undergo three stages, including linear elastic, pseudo-hardening, and softening, which is evident in the load and deflection curves obtained, as shown in Figure 7. The RSF and the cement-based matrix underwent elastic behavior in the first stage, with the maximum flexural strength being achieved in this stage attributed to the matrix strength. In turn, the RSF contributed very little to the overall flexural behavior. Microcracks appeared near the notch in the second stage, where the cracking strain of the cement-based matrix was exceeded. As the load increased, the microcracks merged into a single macrocrack above the notch. Crack surfaces were bridged with closely spaced RSF. This microcrack slowly widens at this stage, possibly due to the high strength of RSF and the strong bond between the fibers and the matrix, leading to a certain level of strain-hardening. This is the main characteristic that distinguishes UHPFRC from traditional concrete. This pseudo-strain-hardening stage is the final of the three stages of flexural response. The area under the load versus deflection curve of the ECAT-based UHPFRC samples was observed to be relatively higher than that of the plain UHPFRC samples, indicating that the stiffness of the UHPFRC was improved in the presence of ECAT.

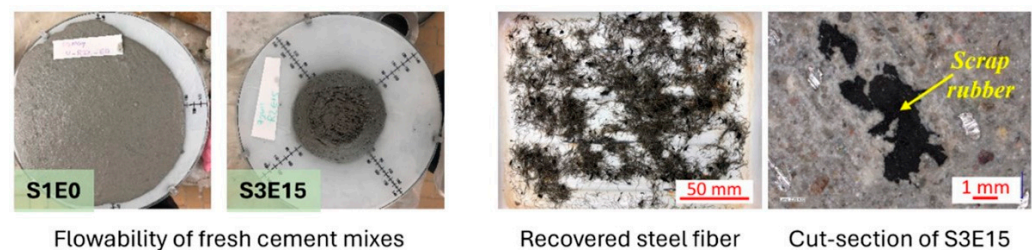


Figure 5. Flowability and cut-section of the UHPFRC samples.

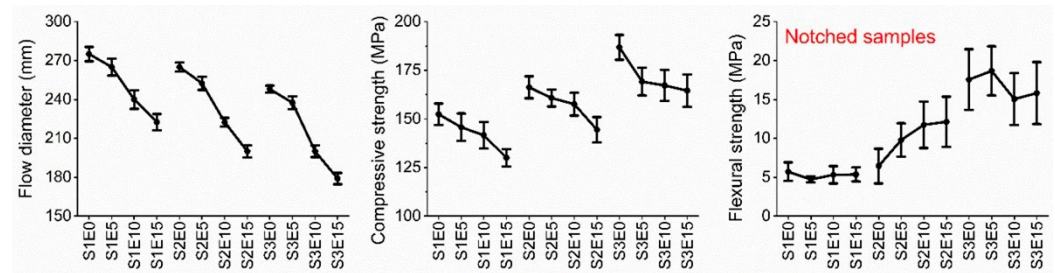


Figure 6. Various properties of the different UHPFRC samples.

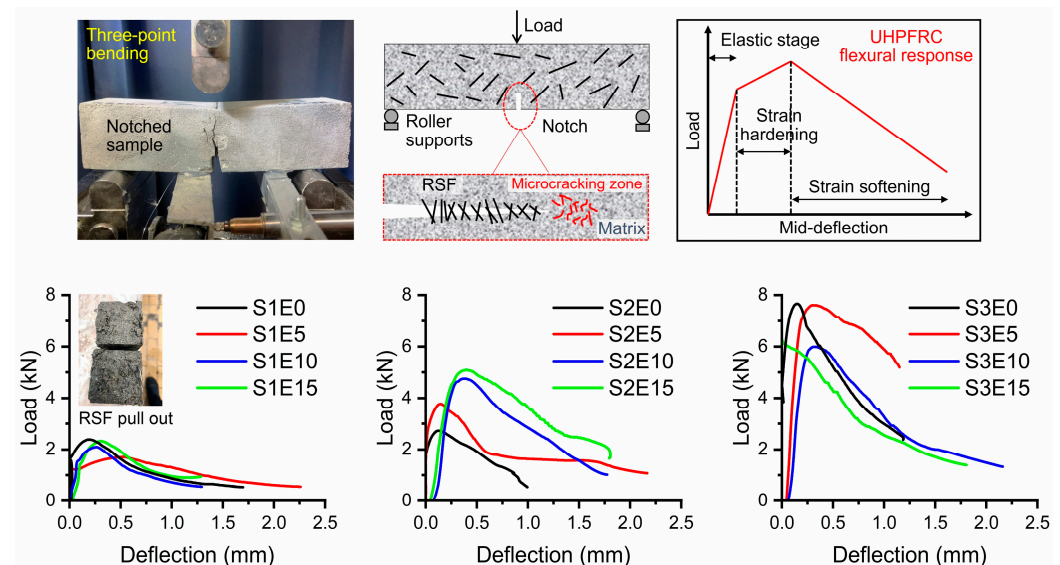


Figure 7. Load and deflection curves obtained by performing the flexural strength test (inset images showing the pull out of RSF in the sample cross-section, the flexural response of a typical UHPFRC sample, and the microcracking zone in the notched region [50]).

3.3. Economic and Environmental Impacts

The assessment of the CO₂ emissions caused by the created UHP-FRCs used the theoretical proposed in Pillai, et al. [51], ISO 14040 [52], and ISO 14044 [53]. Figure 8 provides a schematic illustration of this framework, which consists of four primary steps: the system's scope and restrictions are defined, raw material inventory is gathered, power and fuel consumption are examined, the influence of raw materials on CO₂ emissions and energy demand is assessed, and various raw material impacts are compared. In order to evaluate the environmental impact of cement concrete, mechanical and durability aspects are taken into account. A quality criterion used by the construction industry is the compressive strength of concrete mixes after 28 days. The environmental effects of the UHPFRC were assessed using a similar criterion in this study as well. Figure 9 illustrates the various operations associated with the ground-to-gate process of the developed UHPFRC. The evaluation of the material life cycle made use of information on the carbon impacts related to the different stages of the production of concrete, such as the extraction, production, and transportation of fuel; the extraction and transportation of raw materials needed to make cement; the extraction and transportation of sand; and the infrastructure needed for concrete mixing plants and electricity generation. The energy demand, CO₂ emissions, transportation used, and material and electricity consumption are also illustrated in Figure 9. A typical cement plant in the city of Wroclaw in Poland was recognized as the primary source for obtaining this information. An inventory analysis was also conducted using statistical information obtained from the EcoInvent database, the Intergovernmental Panel on Climate Change report, and the United States Environmental Policy Act (EPA). CO₂ emission is evaluated to assess environmental impacts. Pillai, et al. [51] and

Gettu, et al. [54] listed crucial CO₂ conversion factors, which were used in the environmental impact evaluation of the mixes of UHPC and UHPFRC. Cargo trucks of a total capacity of 23 MT were used during transportation activities. It was calculated how much fuel they used. The EcoInvent database, which typically covers direct and indirect emissions linked to fuel extraction and consumption, vehicle production and maintenance, road building, etc., was used to evaluate the impact of such usage on quantifying CO₂ emissions. The Construction Laboratory of the Department of Civil Engineering at Wroclaw University and Cracow University in Poland produced each of the cement mixes indicated in Table 1. The actual transportation distances measured were 397, 3, 320, 766, 128, and 273 km from this laboratory facility to the supply sources of Portland cement, fly ash, silica fume, ECAT, RSF, and river sand. The impact of the concrete mixing plant is taken from the EcoInvent database. The recent market costs of the raw materials, including cement, silica fume, RSF, river sand, water, and superplasticizer offered by Polish suppliers, are illustrated in Figure 8, but there is only transportation cost involved for fly ash and ECAT because they are solid waste disposed of during the electricity generation and crude oil refining processes. The CO₂ emissions of the cement, silica fume, sand, water, and superplasticizer during their production and extraction were 0.84, 0.014, 0.0024, 0.00015, and 0.00017 kg·CO₂/kg. These values, reported by Mocharla, et al. [55], were used during the UHPFRC environmental impact analysis. CO₂ emissions from industrial by-products and waste materials, including fly ash, ECAT, and RSF, were negligible and therefore considered zero in this environmental impact assessment. The total emissions of CO₂ in kg per m³ of these mixes were calculated using the CO₂ conversion factors listed by Pillai, et al. [51]. Figure 8 shows the material cost and CO₂ emitted by the developed UHPFRCs. There is a gradual decrease in these UHPFRC parameters with an increasing replacement of cement with ECAT. The cost and CO₂ emissions of S0E15 were found to be USD 188 and 585 kg·CO₂ eq./m³ of the mix, and were the lowest among the cement-based mixes that do not contain RSF in them. The addition of 3% RSF to this mix increased the cost by 88%, which was mainly due to the fare involved to transport the RSF from the mechanical separation plants to the concrete manufacturing site. However, there is no change in CO₂ emissions because steel fibers recovered from scrap tires were used instead of traditional steel fibers. The cost of S1E0, S2E0, and S3E0 was determined to be 259, 314, and 369 USD/m³ of mix. Sourcing scrap tires and installing recycling plants locally to recover steel fibers can open a new business market and reduce economic and environmental impacts. The addition of ECAT in the UHPFRC reduced the cost and CO₂ emissions of the UHPFRC by up to 4% and 15%. The presence of ECAT did not affect the flexural strength of the 2% RSF-reinforced UHPFRC samples at early stages. The addition of more RSF increased the mechanical performance of the UHPFRC, but the cost was significantly increased. The Orlen petrochemical company disposes of around 5515 MT of ECAT annually, according to a 2015 survey [56], and the majority of this waste is sent to landfills, causing economic losses and serious health risks related to the release of heavy metal contaminants into the natural environment. The availability of ECAT in Poland is abundant due to the presence of Orlen-owned crude oil refineries in the Baltic Sea, and this waste can be effectively valorized by the Polish construction sector to produce structural concrete mixes for heavy civil infrastructure projects.

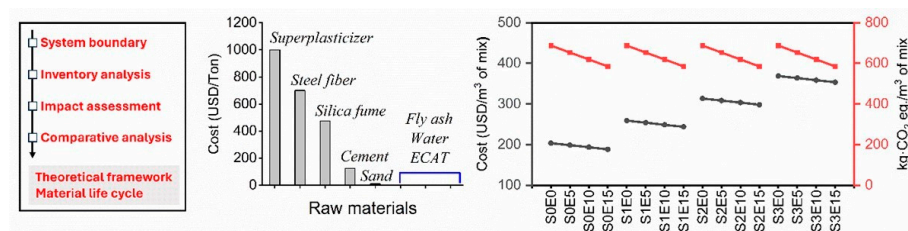


Figure 8. The CO₂ emission and material cost of the developed UHPFRCs.

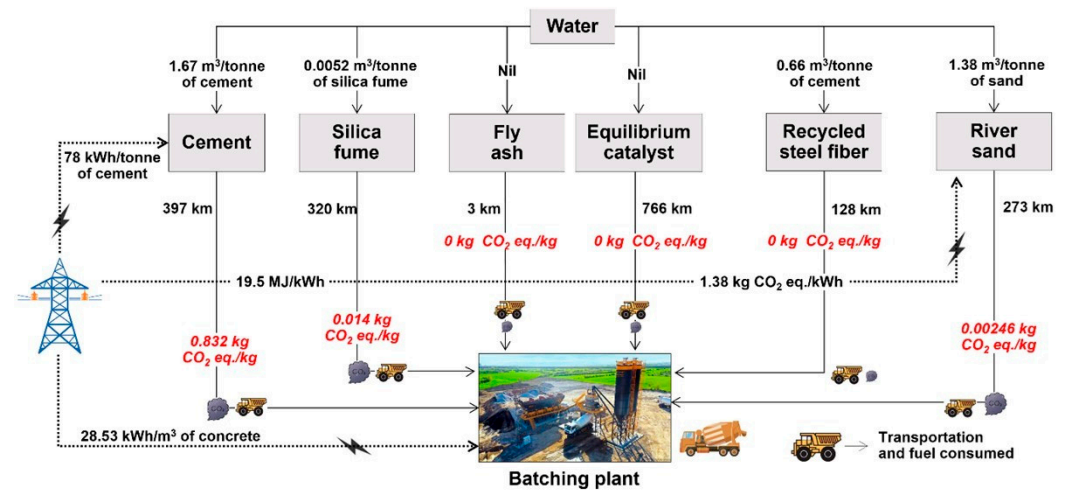


Figure 9. Different operations in the ground-to-gate process of the preparation of the S3E15 mix.

4. Conclusions

In this study, UHPFRC samples containing waste derived from scrap tires and oil refineries were tested. The ECAT particles were found to have a porous microstructure. Their additions absorbed free water on their surfaces and thus reduced the flow of fresh cement mixes by 17%. There is a gradual increase in the compressive and flexural strengths of the UHPFRC samples with an increasing RSF content, indicating the ability of RSF to disperse uniformly in concrete matrices. The replacement of cement with ECAT reduced heat developments by up to 16.5% at the end of the calorimetry test. The compressive strength of the ultrahigh-performance cement-based mix containing silica fume and fly ash was determined to be 155 MPa at the end of 28 days. However, this value was reduced by 17% when the cement was replaced with ECAT by up to 15%. There was no significant change in flexural strength, indicating that the brittleness of the mortar samples was reduced by a partial replacement of cement with spherical ECAT particles. The addition of RSF to the ultrahigh-performance cement-based mixes containing silica fume, fly ash, and ECAT was found to increase compressive strength by 30%. The UHPFRC mixes without any ECAT achieved compressive and flexural strengths of 187 and 26 MPa at the end of 28 days. The UHPFRC samples with a central notch underwent strain-hardening and -softening mechanisms in three-point bending conditions. Increasing ECAT additions in UHPFRC mixes did not affect the flexural behavior of such mixes modified with RSF by up to 2%. In contrast, there was a marginal decrease in the flexural strength of the 3% RSF-reinforced UHPFRC samples with an increasing amount of ECAT. Cement binds RSF and sand together in the UHPFRC matrix, and its replacement with pozzolanic materials only affected the tensile properties in the case of a higher volume of RSF (i.e., 3% by volume). The addition of ECAT reduced the cost and CO₂ emissions of the UHPFRC mixes by 7% and 15%, but its additions at higher doses affected the flow and strength gain properties of the concrete mixes in the early stages. The acquisition of scrap tires and the installation of recycling plants locally to recover steel fibers can open a new business market and significantly reduce economic and environmental impacts. These experimental findings suggest that the development of UHPFRC is feasible on a larger scale with the use of waste derived from scrap tires and crude oil refineries. Moreover, the extensive application of this environmentally friendly UHPFRC in bridge deck construction could help achieve the goal of being carbon neutral.

Author Contributions: Conceptualization, H.A. and M.M.; methodology, H.A. and M.M.; validation, M.M.; formal analysis, M.M.; investigation, H.A., M.M., L.H. and P.K.; resources, L.S. and A.K.; data curation, M.M., L.H. and P.K.; writing—original draft preparation, M.M.; writing—review and editing, P.N., L.S. and A.K.; visualization, M.M.; supervision, L.S.; project administration, H.A. and

M.M.; funding acquisition, H.A. and M.M. All authors have read and agreed to the published version of the manuscript.

Funding: This research was funded by Narodowa Agencja Wymiany Akademickiej (NAWA), grant numbers PPN/ULM/2020/1/00286 and BPN/ULM/2021/1/00120.

Institutional Review Board Statement: Not applicable.

Informed Consent Statement: Not applicable.

Data Availability Statement: The original contributions presented in the study are included in the article, further inquiries can be directed to the corresponding author.

Conflicts of Interest: The authors declare no conflicts of interest.

References

1. You, W.; Bradford, M.A.; Liu, H.; Zhao, W.; Yang, G. Steel-alkali activated cement based ultra-high performance concrete lightweight composite bridge decks: Flexural behavior. *Eng. Struct.* **2022**, *266*, 114639. [\[CrossRef\]](#)
2. Kromoser, B.; Preinstorfer, P.; Kollegger, J. Building lightweight structures with carbon-fiber-reinforced polymer-reinforced ultra-high-performance concrete: Research approach, construction materials, and conceptual design of three building components. *Struct. Concr.* **2019**, *20*, 730–744. [\[CrossRef\]](#)
3. Huang, Y.; Grünwald, S.; Schlangen, E.; Luković, M. Strengthening of concrete structures with ultra high performance fiber reinforced concrete (UHPFRC): A critical review. *Constr. Build. Mater.* **2022**, *336*, 127398. [\[CrossRef\]](#)
4. Yin, T.; Liu, K.; Fan, D.; Yu, R. Derivation and verification of multilevel particle packing model for ultra-high performance concrete (UHPC): Modelling and experiments. *Cem. Concr. Compos.* **2023**, *136*, 104889. [\[CrossRef\]](#)
5. Akeed, M.H.; Qaidi, S.; Ahmed, H.U.; Faraj, R.H.; Mohammed, A.S.; Emad, W.; Tayeh, B.A.; Azevedo, A.R.G. Ultra-high-performance fiber-reinforced concrete. Part I: Developments, principles, raw materials. *Case Stud. Constr. Mater.* **2022**, *17*, e01290. [\[CrossRef\]](#)
6. Niewiadomski, P.; Karolak, A.; Oleksiak, M.; Chowaniec-Michalak, A. The effectiveness of impregnation of building materials of cellulose origin and its impact on selected properties. *Bull. Pol. Acad. Sci. Tech. Sci.* **2024**, *72*, e148876. [\[CrossRef\]](#)
7. Chu, S.H.; Li, L.; Shen, P.L.; Lu, J.X.; Poon, C.S. Recycling of waste glass powder as paste replacement in green UHPFRC. *Constr. Build. Mater.* **2022**, *316*, 125719. [\[CrossRef\]](#)
8. Abdolpour, H.; Niewiadomski, P.; Sadowski, Ł. Recycling of steel fibres and spent equilibrium catalyst in ultra-high performance concrete: Literature review, research gaps, and future development. *Constr. Build. Mater.* **2021**, *309*, 125147. [\[CrossRef\]](#)
9. Martin, C.; Manu, E.; Hou, P.; Adu-Amankwah, S. Circular economy, data analytics, and low carbon concreting: A case for managing recycled powder from end-of-life concrete. *Resour. Conserv. Recycl.* **2023**, *198*, 107197. [\[CrossRef\]](#)
10. Guo, Y.; Luo, L.; Liu, T.; Hao, L.; Li, Y.; Liu, P.; Zhu, T. A review of low-carbon technologies and projects for the global cement industry. *J. Environ. Sci.* **2024**, *136*, 682–697. [\[CrossRef\]](#)
11. Matos, A.M.; Nunes, S.; Costa, C.; Aguiar, J.L.B. Durability of an UHPC containing spent equilibrium catalyst. *Constr. Build. Mater.* **2021**, *305*, 124681. [\[CrossRef\]](#)
12. Payá, J.; Monzó, J.; Borrachero, M.V.; Velázquez, S. Cement equivalence factor evaluations for fluid catalytic cracking catalyst residue. *Cem. Concr. Compos.* **2013**, *39*, 12–17. [\[CrossRef\]](#)
13. Allahverdi, A.; Vakili, S.; Gharabeglu, P. Effects of RFCC spent catalyst on some physicomechanical properties of portland cement paste. *Ceram. Silikáty* **2011**, *55*, 161–168.
14. Nunes, S.; Costa, C. Numerical optimization of self-compacting mortar mixture containing spent equilibrium catalyst from oil refinery. *J. Clean. Prod.* **2017**, *158*, 109–121. [\[CrossRef\]](#)
15. Matos, A.M.; Nunes, S.; Costa, C.; Barroso-Aguiar, J.L. Spent equilibrium catalyst as internal curing agent in UHPFRC. *Cem. Concr. Compos.* **2019**, *104*, 103362. [\[CrossRef\]](#)
16. Payá, J.; Monzó, J.; Borrachero, M.V.; Velázquez, S.; Bonilla, M. Determination of the pozzolanic activity of fluid catalytic cracking residue. Thermogravimetric analysis studies on FC3R–lime pastes. *Cem. Concr. Res.* **2003**, *33*, 1085–1091. [\[CrossRef\]](#)
17. Harenberg, S.; Caggiano, A.; Koenig, A.; Said, D.; Gilka-Bötzow, A.; Schultz-Cornelius, M.; Yang, S.; Pahn, M.; Dehn, F.; Koenders, E. Micromechanical behavior of UHPC under cyclic bending-tensile loading in consideration of the influence of the concrete edge zone. *PAMM* **2018**, *18*, e201800363. [\[CrossRef\]](#)
18. Koorikkattil, A.; Nayar, S.K.; Venudharan, V. Flexural fatigue design of steel fibre reinforced concrete pavements: A comparative study. *Road Mater. Pavement Des.* **2023**, *24*, 2301–2321. [\[CrossRef\]](#)
19. Qin, X.; Kaewunruen, S. Environment-friendly recycled steel fibre reinforced concrete. *Constr. Build. Mater.* **2022**, *327*, 126967. [\[CrossRef\]](#)
20. Kundak, M.; Lazić, L.; Črnko, J. CO₂ emissions in the steel industry. *Metalurgija* **2009**, *48*, 5.
21. Zaid, O.; Mukhtar, F.M.; M-García, R.; El Sherbiny, M.G.; Mohamed, A.M. Characteristics of high-performance steel fiber reinforced recycled aggregate concrete utilizing mineral filler. *Case Stud. Constr. Mater.* **2022**, *16*, e00939. [\[CrossRef\]](#)

22. Wang, T.; Yu, M.; Shan, W.; Xu, L.; Cheng, S.; Li, L.-y. Post-fire compressive stress–strain behaviour of steel fibre reinforced recycled aggregate concrete. *Compos. Struct.* **2023**, *309*, 116735. [[CrossRef](#)]
23. Mastali, M.; Dalvand, A. Fresh and Hardened Properties of Self-Compacting Concrete Reinforced with Hybrid Recycled Steel–Polypropylene Fiber. *J. Mater. Civ. Eng.* **2017**, *29*, 04017012. [[CrossRef](#)]
24. Martinelli, E.; Caggiano, A.; Xargay, H. An experimental study on the post-cracking behaviour of Hybrid Industrial/Recycled Steel Fibre-Reinforced Concrete. *Constr. Build. Mater.* **2015**, *94*, 290–298. [[CrossRef](#)]
25. Neocleous, K.; Angelakopoulos, H.; Pilakoutas, K.; Guadagnini, M. Fibre-reinforced roller-compacted concrete transport pavements. *Proc. Inst. Civ. Eng. Transp.* **2011**, *164*, 97–109. [[CrossRef](#)]
26. Aghaee, K.; Yazdi, M.A.; Tsavdaridis, K.D. Investigation into the mechanical properties of structural lightweight concrete reinforced with waste steel wires. *Mag. Concr. Res.* **2015**, *67*, 197–205. [[CrossRef](#)]
27. Peng, G.; Yang, J.; Long, Q.; Niu, X.; Shi, Y. Mechanical properties and explosive spalling behavior of the recycled steel fiber reinforced ultra-high-performance concrete. In *Multi-Span Large Bridges*; CRC Press: Boca Raton, FL, USA, 2015; pp. 1039–1048.
28. Krassowska, J.; Kosior-Kazberuk, M. Experimental investigation of shear behavior of two-span fiber reinforced concrete beams. *Arch. Civ. Eng.* **2019**, *65*, 35–55. [[CrossRef](#)]
29. Zamanzadeh, Z.; Lourenço, L.; Barros, J. Recycled Steel Fibre Reinforced Concrete failing in bending and in shear. *Constr. Build. Mater.* **2015**, *85*, 195–207. [[CrossRef](#)]
30. Graeff, A.G.; Pilakoutas, K.; Neocleous, K.; Peres, M.V.N.N. Fatigue resistance and cracking mechanism of concrete pavements reinforced with recycled steel fibres recovered from post-consumer tyres. *Eng. Struct.* **2012**, *45*, 385–395. [[CrossRef](#)]
31. Isa, M.N.; Pilakoutas, K.; Guadagnini, M.; Angelakopoulos, H. Mechanical performance of affordable and eco-efficient ultra-high performance concrete (UHPC) containing recycled tyre steel fibres. *Constr. Build. Mater.* **2020**, *255*, 119272. [[CrossRef](#)]
32. Le, H.V.; Dao, P.L.; Nguyen, S.D.; Ngo, T.T.; Tran, N.T.; Nguyen, D.L.; Kim, D.J. Improvement of the stress sensing ability of ultra-high-performance concrete using short steel fibers and steel slag aggregates under high compression. *Sens. Actuators A Phys.* **2023**, *362*, 114616. [[CrossRef](#)]
33. Abdolpour, H.; Niewiadomski, P.; Kwiecień, A.; Tekieli, M.; Sadowski, Ł. Fundamental understanding of the mechanical and post-cracking behaviour of ultra-high performance mortar with recycled steel fibers. *Constr. Build. Mater.* **2023**, *374*, 130918. [[CrossRef](#)]
34. EN 197-1; Cement: Composition, Specifications and Conformity Criteria for Common Cements. British Standard Institution: London, UK, 2011; p. 46.
35. EN 1097-6; Tests for Mechanical and Physical Properties of Aggregates—Part 6: Determination of Particle Density and Water Absorption. British Standard Institution: London, UK, 2022; p. 61.
36. Yu, R.; Spiesz, P.; Brouwers, H.J.H. Mix design and properties assessment of Ultra-High Performance Fibre Reinforced Concrete (UHPFRC). *Cem. Concr. Res.* **2014**, *56*, 29–39. [[CrossRef](#)]
37. ASTM C1702; Standard Test Method for Measurement of Heat of Hydration of Hydraulic Cementitious Materials Using Isothermal Conduction Calorimetry. ASTM: West Conshohocken, PA, USA, 2017; p. 9.
38. ASTM C230; Standard Specification for Flow Table for Use in Tests of Hydraulic Cement. ASTM: West Conshohocken, PA, USA, 2021; p. 7.
39. EN 12390-3; Testing Hardened Concrete—Part 3: Compressive Strength of Test Specimens. British Standard Institution: London, UK, 2019; p. 9.
40. ASTM C348; Standard Test Method for Flexural Strength of Hydraulic-Cement Mortars. ASTM: West Conshohocken, PA, USA, 2021; p. 6.
41. Yogendran, V.; Langan, B.W.; Ward, M.A. Hydration of cement and silica fume paste. *Cem. Concr. Res.* **1991**, *21*, 691–708. [[CrossRef](#)]
42. Skibsted, J.; Snellings, R. Reactivity of supplementary cementitious materials (SCMs) in cement blends. *Cem. Concr. Res.* **2019**, *124*, 105799. [[CrossRef](#)]
43. Rodríguez, E.D.; Bernal, S.A.; Provis, J.L.; Gehman, J.D.; Monzó, J.M.; Payá, J.; Borrachero, M.V. Geopolymers based on spent catalyst residue from a fluid catalytic cracking (FCC) process. *Fuel* **2013**, *109*, 493–502. [[CrossRef](#)]
44. Ndahirwa, D.; Zmamou, H.; Lenormand, H.; Leblanc, N. The role of supplementary cementitious materials in hydration, durability and shrinkage of cement-based materials, their environmental and economic benefits: A review. *Clean. Mater.* **2022**, *5*, 100123. [[CrossRef](#)]
45. da Cunha, A.L.C.; Lemos, M.S.; Meth, S.; Gonçalves, J.P.; Dweck, J. A study of the particle size effect on the pozzolanic activity of an equilibrium catalyst. *J. Therm. Anal. Calorim.* **2011**, *106*, 805–809. [[CrossRef](#)]
46. Antiohos, S.; Tsimas, S. Investigating the role of reactive silica in the hydration mechanisms of high-calcium fly ash/cement systems. *Cem. Concr. Compos.* **2005**, *27*, 171–181. [[CrossRef](#)]
47. Biswas, R.K.; Bin Ahmed, F.; Haque, M.E.; Provash, A.A.; Hasan, Z.; Hayat, F.; Sen, D. Effects of steel fiber percentage and aspect ratios on fresh and harden properties of ultra-high performance fiber reinforced concrete. *Appl. Mech.* **2021**, *2*, 501–515. [[CrossRef](#)]
48. Abdolpour, H.; Muthu, M.; Niewiadomski, P.; Sadowski, Ł.; Hojdy, Ł.; Krajewski, P.; Kwiecień, A. Performance and life cycle of ultra-high performance concrete mixes containing oil refinery waste catalyst and steel fibre recovered from scrap tyre. *J. Build. Eng.* **2023**, *79*, 107890. [[CrossRef](#)]
49. Lepech, M.D.; Li, V.C. *Size Effect in ECC Structural Members in Flexure*; Aedificatio Publishers: Freiburg, Germany, 2004.

50. Nguyen, D.-L.; Le, H.-V.; Vu, T.-B.-N.; Nguyen, V.-T.; Tran, N.-T. Evaluating fracture characteristics of ultra-high-performance fiber-reinforced concrete in flexure and tension with size impact. *Constr. Build. Mater.* **2023**, *382*, 131224. [[CrossRef](#)]
51. Pillai, R.G.; Gettu, R.; Santhanam, M.; Rengaraju, S.; Dhandapani, Y.; Rathnarajan, S.; Basavaraj, A.S. Service life and life cycle assessment of reinforced concrete systems with limestone calcined clay cement (LC3). *Cem. Concr. Res.* **2019**, *118*, 111–119. [[CrossRef](#)]
52. *ISO 14040; Environmental Management—Life Cycle Assessment—Principles and Framework*. International Organization for Standardization: Geneva, Switzerland, 2006; p. 20.
53. *ISO 14044; Environmental Management—Life Cycle Assessment—Requirements and Guidelines*. International Organization for Standardization: Geneva, Switzerland, 2006; p. 46.
54. Gettu, R.; Patel, A.; Rathi, V.; Prakasan, S.; Basavaraj, A.S.; Palaniappan, S.; Maity, S. Influence of supplementary cementitious materials on the sustainability parameters of cements and concretes in the Indian context. *Mater. Struct.* **2019**, *52*, 10. [[CrossRef](#)]
55. Mocharla, I.R.; Selvam, R.; Govindaraj, V.; Muthu, M. Performance and life-cycle assessment of high-volume fly ash concrete mixes containing steel slag sand. *Constr. Build. Mater.* **2022**, *341*, 127814. [[CrossRef](#)]
56. Niewiadomski, P.; Cisiński, M. The impact of waste fluid catalytic cracking catalyst addition on the selected properties of cement pastes. *Mater. Proc.* **2023**, *13*, 10. [[CrossRef](#)]

Disclaimer/Publisher’s Note: The statements, opinions and data contained in all publications are solely those of the individual author(s) and contributor(s) and not of MDPI and/or the editor(s). MDPI and/or the editor(s) disclaim responsibility for any injury to people or property resulting from any ideas, methods, instructions or products referred to in the content.