

Design of an innovative self-compacting material modified with recycled steel fibers and spent equilibrium catalyst for ultra-high performance applications

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Abstract

The main aim of the present study is to design an innovative self-compacting material modified with recycled steel fibers (RSF) from waste tires and spent equilibrium catalyst (Ecat) from the petrochemical industry for ultra-high performance application. For this purpose, 17 different mixtures were developed and analysed using different percentages of RSF (0%-3%) and replacement of cement by different percentages of Ecat (0%-15%).

The developed mortars' self-compatibility was evaluated in a fresh stage using mini-cone tests. Regarding the hardened stage, the mortars were characterized at the ages of 7 days and 28 days using compression and unnotched flexural tests. The abilities of RSF to increase the post-cracking behavior of the specimens and to use Ecat to increase the bond performance between RSF and the cement matrix were assessed by performing notched three-point bending tests. The results of notched flexural tests were used to obtain the residual flexural strength in service limit state (SLS), ultimate limit state (ULS), and two equivalent flexural strengths.

The experimental results for the fresh stage demonstrated that inclusion of RSF and Ecat significantly reduced the workability of mortars. It was observed that using RSF and Ecat resulted in increasing compressive strength and flexural strength of specimens. Notched flexural tested specimens showed that the addition of RSF and Ecat can significantly decrease the brittle behavior of cement-based materials by improving its toughness and post-cracking resistance. Middle-span deflection, crack initiation load, and ultimate flexural load were also increased with the addition of RSF and Ecat. In this sense, the results of this research showed that RSF and Ecat seem to have the potential to constitute a sustainable material for structural and nonstructural applications.

Author Keywords. Ultra-high Performance Mortar, Self-Compacting, Recycled Steel Fiber, Spent Catalyst, Residual Flexural Strength

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Introduction

At the beginning of the 1990s, a new form of cement-based composite materials was invented (De Larrard and Sedran 1994) using a low water to binder ratio and refine admixture nominated by ultra-high performance fiber reinforced concrete (UHPFRC). The constituents of ultra high performance concrete (UHPC) are close to conventional concrete using Portland cement, silica fume, limestone filler, fine sand, water reducer admixture, steel fiber, etc. (Kono

et al. 2013). This grade of concrete is characterized by two main factors, namely (i) outstanding mechanical properties and (ii) higher durability properties (De Larrard and Sedran 1994). Combination of these two factors makes UHPC an ideal candidate for use in applications in such harsh marine environments where longer service life and less maintenance are the main demands (Kono et al. 2013).

Currently, energy and raw materials consumptions are becoming restricted, and many construction companies are paying more attention to the use of sustainable materials. Consequently, despite the excellent mechanical properties of UHPFRC, it is still considered inconsistent due to the higher cost of production (due to higher price for the steel fiber) and CO₂ emissions (due to higher content of the cement). For example, almost two thirds of the price of final production is related to the price of steel fiber (Matos et al. 2019). Furthermore, it is recommended to use 800-1000 kg/m³ (W. Peng and Sui Pheng 2011) of cement, which is approximately three times the amount of cement in normal concrete (Richard and Cheyrezy 1995) and requires high energy consumption and CO₂ emissions. This implies that UHPFRC needs to be further optimized and its final cost and ecological developments require more investigation, which is the main inherent of this research.

Disposal of construction waste has been studied in the literature widely. However, using petrochemical waste in the construction field has a great potential to be replaced by raw materials. In oil refinery factories, one of the indispensable materials in a powder shape is fluid cracking catalyst (FCC), which is vital for breaking the molecules of high-weight crude oils. Since the cracking process requires high temperatures thereafter, the FCC becomes disactivated, which should be replaced by the new one. The proposed spent catalyst (Ecat) is a waste material which mostly is subjected to be disposed in landfills. It was testified that Ecat was characterized by pozzolanic activity. This pozzolanic activity makes Ecat more beneficial for the production of cementitious composites by reacting with the calcium hydroxide (CH) (Payá et al. 2013) produced during the hydration of Portland cement. The benefits of using Ecat in the production of cementitious products such as paste (Payá et al. 2013), mortar (Allahverdi, Vakilinia, and Gharabeglu 2011), self-compacting concrete (Nunes and Costa 2017) and ultra UHPFRC (Matos et al. 2019) were presented in the literature. The increase in mechanical properties can be justified by the fact that the reaction of Ecat with CH and could result in the production of calcium silicate hydrate (CSH), calcium aluminate hydrate (CAH), and calcium aluminosilicate hydrate (CASH), which are fundamental in enhancing the mechanical strength of cementitious materials (Payá et al. 2003). With respect to the workability decrease, the main justification for this fact is that the Ecat, due to large BET (Brunauer-Emmett-Teller) surface area and highly porous microstructure, increases the potentiality for high capacity of water absorption.

Reinforcement of cementitious matrix using discontinuous fibers is a widely recognized technology to enhance mechanical properties, including post-cracking behaviour of materials. After initiation of the crack, the fibers exert to arrest the cracks using a bridging action and to improve the post-peak and residual strength of the element (G. F. Peng et al. 2015; Leone et al. 2018). The initial high cost of UHPFRC is due to the use of steel fibers. For example, the price of conventional concrete is about 200 \$ / m³, while in the case of using UHPFRC this price can be almost 5 times higher (Graybeal 2013). In addition to the cost of steel fiber, sustainability problems of steel production should be taken into account. Each ton of steel was estimated to emit 1.9 tons of CO₂ (Kundak, Lazić, and Črnko 2009). Therefore, substituting steel fibers with other alternative recycled steel fibers (RSF) could be beneficial in terms of reducing the initial cost and increasing the sustainability of UHPFRC. The effects of the

addition of RSF on the fresh properties and hardened properties of high-performance concrete (HPC) have been thoroughly studied in the literature (Liew and Akbar 2020). It was shown that increasing the quantity of RSF not only adversely affects the workability but also the influence of the homogeneity of the fresh mixture. A decrease in compressive strength was reported from 135.5 MPa to 130.2 MPa in the case of fibers with rubbers attached, while a decrease of 5% of compressive strength was reported at 141.30 MPa in the case of fibers without any rubbers attached (Mastali and Dalvand 2017). In the case of flexural toughness reinforced by HPC following RSF, a gradual increase in post-peak fiber percentage was reported (Aghaee, Yazdi, and Tsavdaridis 2015). A relatively smaller amount of residual flexural strength and softening responses was revealed at a lower content of fiber utilization, while a significant improvement in flexural strength was observed when the amount of fiber increased (Zamanzadeh, Lourenço, and Barros 2015). Based on that the main inherent aim of this study is investigation for producing some self-compacting ultra-high performance mortars using recycled steel fiber and spent catalyst.

1. Materials and methods

1.1. Materials

The raw materials used in this research were used from local available gradients in Poland, which include Ordinary Portland Cement (OPC-Type II) produced by Royal Cement (Pszczółki, Poland), silica fume (SF) produced by Mikrosilika Trade, fly ash (FA), spent catalyst (Ecat) produced by ORLEN, sand (S) produced by Trzuskawica, water (w) and superplasticizer (SP) produced by Sika (ViscoCrete-93 RS) in a liquid form. For determining the chemical compositions of the raw materials, the Z-ray fluorescence technique was used, and the results are tabulated in Table 1.

Substance (%)	Mass fraction of the sample (%)								
	MgO	Al ₂ O ₃	SiO ₂	SO ₃	K ₂ O	CaO	Na ₂ O	Fe ₂ O ₃	Other
OPC	0.54	2.14	17.14	6.94	0.89	38.13	---	---	48.94
SF	---	---	39.54	---	---	--	---	---	60.46
FA	2.67	26.23	48.17	---	5.52	5.16	2.61	9.95	---
Ecat	3.43	38.11	54.58	3.88	---	---	---	---	---
S	---	---	45.08	---	---	---	---	---	54.92

OPC: Cement (type II- 52.5 R), SF: Silica fume, fa: fly ash, Ecat: spent catalyst, S:sand

Table 1. Chemical composition of the proposed mortar

1.2. Mix proportion and regime

In this study, 16 mixes were designed as shown in Table 2. The nomenclature was used to identify mixes can be explained as: The first character is a letter representing ultra-high performance mortar; the second character is a letter representing reinforcement of ultra high performance mortar (UHPM). The number allocated for this letter designated recycled steel fiber volume fraction. For instance R3% means that the volume fraction of fiber is 3 percent. The third letter represents the Ecat. The number of this letter is designated for the amount of Ecat utilized. For instance, E5% means that 5% of cement was substituted by Ecat. Accordingly, U-R2%-E15% means that the developed ultra-high performance mortar contains 2% of RSF and 115% of cement (by the weight) was replaced by Ecat. Six groups of mixtures were defined. The first one is related to the reference UHPM without considering the addition of

RSF and/or Ecat. The second group belongs to UHPM where RSF was not used and the cement content was substituted by 5%, 10% and 15% by Ecat. The third group of mixtures related to the mixtures in which only the RSF content was varied between 1% and 3% and Ecat was used in this group. The fourth group, fifth group and sixth group of mixes, Ecat was constant in each group by the values of 5%, 10% and 15% while RSF was varied between 1% and 3%.

Group	Notation	OPC	SF	FA	W	S	SP	Ecat	RSF
Group1	U-R0-E0	820	190	150	181.8	1068	25	0	0
Group 2	U-R0-E5	779	190	150	181.8	1068	25	41	0
	U-R0-E10	738	190	150	181.8	1068	25	82	0
	U-R0-E15	697	190	150	181.8	1068	25	123	0
Group 3	U-R1-E0	820	190	150	181.8	1068	25	0	78.5
	U-R2-E0	820	190	150	181.8	1068	25	0	157.0
	U-R3-E0	820	190	150	181.8	1068	25	0	235.5
Group 4	U-R1-E5	779	190	150	181.8	1068	25	41	78.5
	U-R2-E5	779	190	150	181.8	1068	25	41	157.0
	U-R3-E5	779	190	150	181.8	1068	25	41	235.5
Group 5	U-R1-E10	738	190	150	181.8	1068	25	82	78.5
	U-R2-E10	738	190	150	181.8	1068	25	82	157.0
	U-R3-E10	738	190	150	181.8	1068	25	82	235.5
Group 6	U-R1-E15	697	190	150	181.8	1068	25	123	78.5
	U-R2-E15	697	190	150	181.8	1068	25	123	157.0
	U-R3-E15	697	190	150	181.8	1068	25	123	235.5

OPC: Cement (type II- 52.5 R), SF: Silica fume, FA: fly ash, W: water, S: sand, SP: superplasticizer, Ecat: spent catalyst, RSF: recycled steel fiber

Table 2. Compositions of the prepared mixtures (kg/m³)

All of the proposed mixtures were prepared in batches of 2.0 liter volume following six regimes, with the constant speed of mixer (140 rpm). For starting, all the dry components, except than sand and RSF were mixed for a duration of 5 minutes (stage 1). After 5 minutes, half of the water and superplasticizer were gradually added to the mixture for 2 minutes (stage 2). Subsequently, the mixing process was stopped, and all adherent components were removed from the bowels for 1 minute (stage 3). The mixing process continued by pouring the rest of superplasticizer and water into the mixture for 2 minutes (stage 4). Sand and RSF were added to the mixture for 4 minutes. After finishing the process of the mixture, the mortar was directly filled inside the prismatic molds (40 mm × 40 mm × 160 mm) without applying any vibration based on self-compatibility. The samples were consolidated and covered with plastic sheets and after 24 hours demolded and placed inside the curing tank at a water temperature of 20±2°C for the age of the test.

1.3. Testing procedure

1.3.1. Fluidity

The fluidity of the mortars was assessed according to EFNARC (Graeff et al. 2012) using a mini cone (100mm in diameter at the base, 70mm in diameter at the top and 60mm in height). The

cone was filled using fresh mortar and after consolidation lifted up without any shaking that influenced the fluidity. The fluidity of the mortars was measured using the average of the two orthogonal diameters in each developed mixes.

1.3.2. Mechanical performance

In addition to that, the mechanical performances of mortars were assessed in terms of compression strength using specimens with geometric shapes of 80 mm × 40 mm × 40 mm and flexural strength tests using specimens with geometric shapes of 160 mm × 40 mm × 40 mm. For compressive strength, half of the samples at 7 and 28 days after performing flexural tests were subjected to uniaxial compression tests according to ASTM C349-08 (EFNARC 2002) using an electromagnetic universal testing machine (Tecnotest KI 300) with a load cell capacity of 300 kN and a force control with a constant rate of 0.50 N/mm²/s (Fig. 1a).

The flexural behaviour of specimens was assessed. The unnotched prismatic beams were tested at the age of 7 days and 28 days under three point bending tests following the standard ASTM C78 (ASTM 2008). Universal testing machine with a load cell capacity of 300 kN using the force control with a constant rate of 0.12 N/mm²/s was employed. Where the load was applied in the middle of the span and the supports were distanced by a value of 100 mm (Fig. 1b).

To understand the influences of RSF and Ecat on the post-cracking behavior of developed mortars, three-point bending tests were performed on notched specimens at the age of 28 days following RILEM T. 162-TDF (ASTM 2000). To this end, a notched was created using a diamond grinding wheel on the middle bottom side of the specimens having a width of 2 mm and a depth of 10 mm. For the tests, the universal testing machine Zwick 1456 was used using a load cell with a capacity of 20 kN. The specimens were supported by two rigid steel semi cylinders spaced 120 mm and the load was applied at the middle span of the specimens with a constant displacement of 0.5 mm/min (Fig. 1c). Two Linear Variable Displacement Transformers (LVDT) with a 10 mm stroke were used for instrumentation, where one of them was installed vertically to measure vertical deflection and another horizontally to measure crack mouth opening displacement (CMOD).

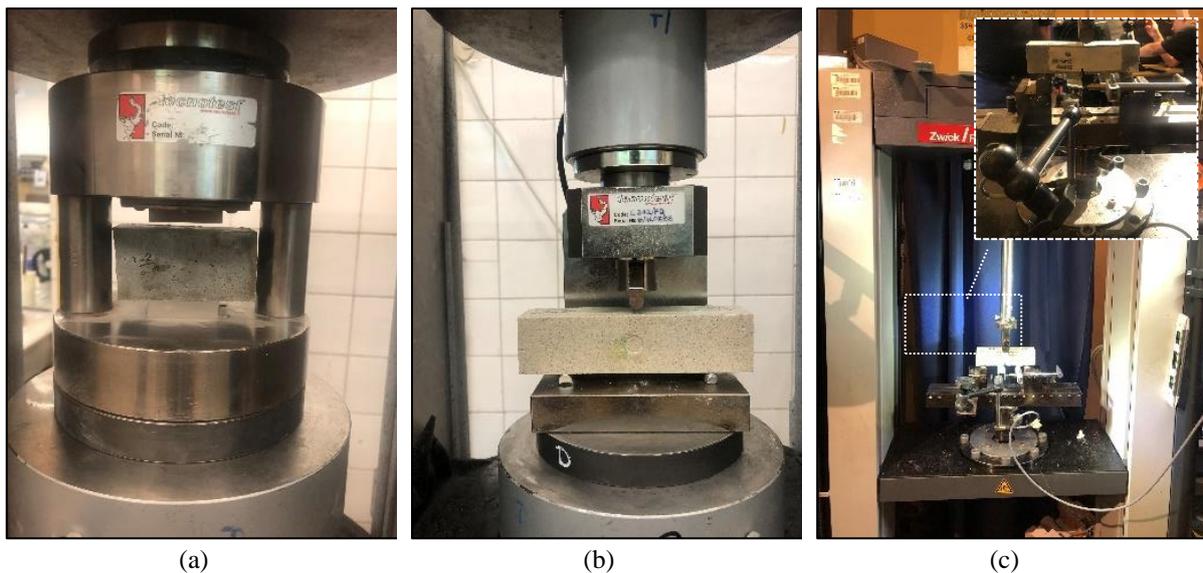


Figure 1. Mechanical performance evaluation of the developed mortars: (a) compression strength, (b) notched flexural strength, (c) notched flexural strength

2. Results and discussion

2.1. Effects of RSF and Ecat on Fluidity

The workability for the developed mortars is presented in Fig. 2. The reference mortar U-R0-E0 is provided for further understanding of the effects of adding Ecat and RSF. The main requirement is also provided based on the criteria proposed in the literature (Vandewalle et al. 2002) for assessing workability. The inspection of the workability showed that all the mixes developed with more than 200 mm had adequate flow and meet the UHPM requirement in the literature (Vandewalle et al. 2002). As expected, using Ecat due to the higher density and pozzolanic activity resulted in decreasing the workability of the mixes. Similarly, in the case of adding RSF to the mixes, it can be seen that when the volume of fiber was increased, the workability of the mixes decreased because of the increased friction between the fibers and pastes. For instance, in Fig.2b it can be observed that, using 1%, 2%, and 3% of RSF workability compared to the reference mortar, was decreased by the percentages of 6.6%, 11.7%, and 17.3% respectively.

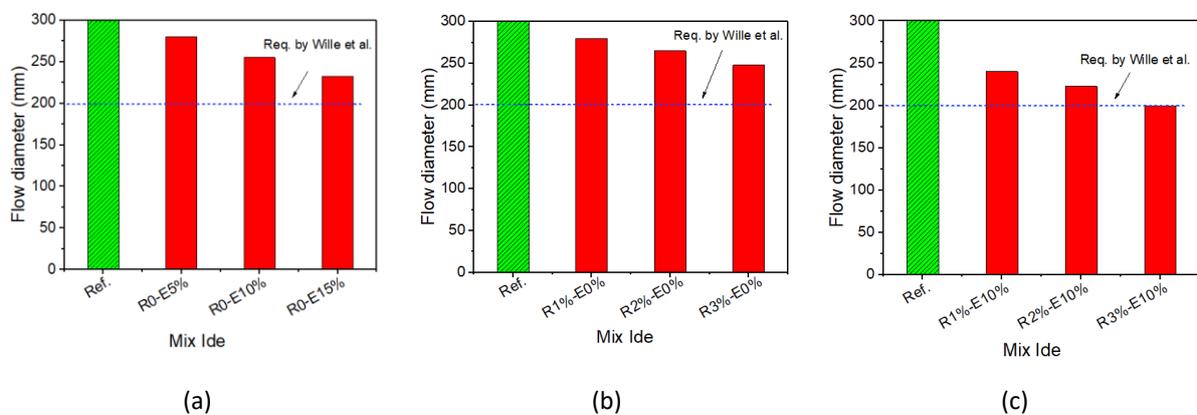


Figure 2. Workability of UHPM mixes: (a) group 2, (b) group 3, (c) group 5

2.2. Mechanical Properties

The compressive strength results for the mixes developed presented in Fig. 3. The results were grouped into two cases for further understanding. In Fig. 3a, the mixes were grouped according to the amount of cement replaced by Ecat. In these groups, the effect of adding different content of RSF on the 7-day compressive strength was presented. Fig. 3b, is presented and the mixes were grouped based on the RSF content, which was varied between 0% and 3%. In this figure, the effects of replacing cement with Ecat, which was varied between 0% and 15%, are presented.

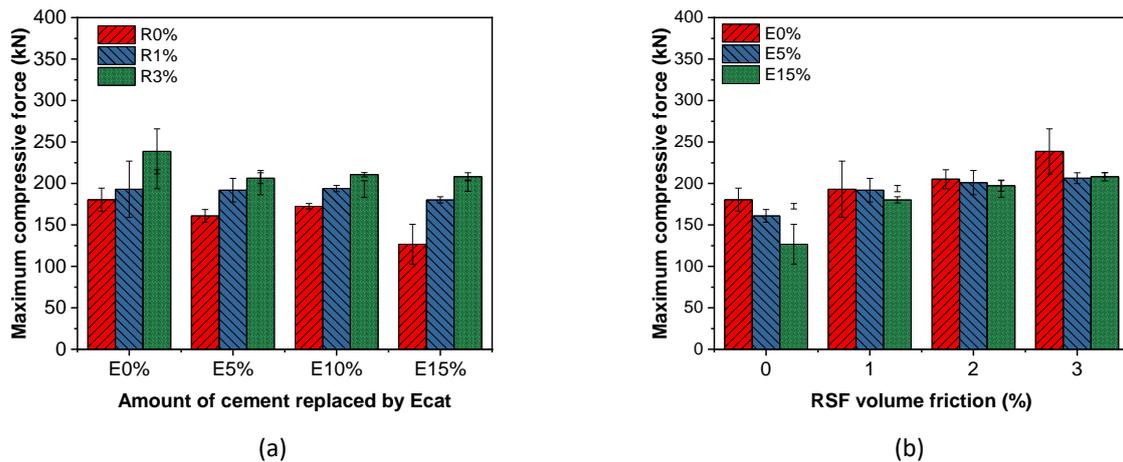


Figure 3. Maximum compressive force of seven days: (a) effects of RSF in different Ecat content, (b) effects of Ecat in different RSF content

Inspection of Figure 3a shows that, in all developed mortars, adding RSF resulted in increasing compressive strength of mixes. This can be explained by the ability of the proposed fiber to crack-stop and, consequently, fibers to bridge actions in increasing compressive strength of the developed mixes. In the case of E0% (without presence of Ecat) adding 1%, 2%, and 3% of RSF compressive strength was increased by 8%, 13% and 32%, respectively, compared with the control mix (U-R0%-E0%).

Figure 3b shows the effects of replacing cement by Ecat in different percentage of RSF. It can be seen that, in the case of unreinforced mixes where RSF was not used until 10% replacement of cement by Ecat, it did not have any influence in decreasing the compressive strength of the tested specimens. In addition to that, it can be seen that replacing the cement replacement with Ecat and using RSF resulted in the compressive strength being nearly constant without a significant decrease. For example, in the case of RSF 3%, the maximum compressive forces in the cases of 5%, 10%, and 15% decreasing of cement was nearly 206 kN.

The maximum compressive force for the 28 days tested specimens is depicted in Fig. 4. Inspection of this figure showed that the effects of using Ecat in 28 days are more pronounced compared to the 7 days tested specimens. In all cases, increasing compressive strength was noticed. It was concluded that until 10% of the replacement of the cement by Ecat the compressive strength was increased. However, after that, a decrease of compressive strength was observed in all cases of specimens with different RSF volume of frictions.

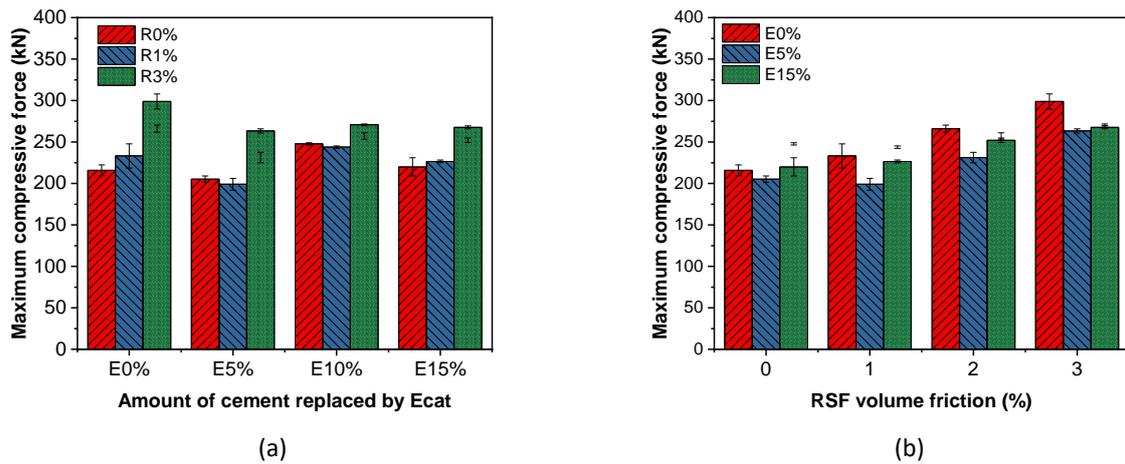


Figure 4. 28 days maximum compressive force: (a) effects of RSF in different content of Ecat, (b) effects of Ecat in different RSF content

In the case of flexural strength of tested mortars, the results are shown in Fig. 5. Fig. 5a shows that in the case of mixes where Ecat was not utilized, adding RSF resulted in increasing flexural capacity of tested beams. It can be seen that the maximum increase in flexural force in the reinforced ultra-high performance mortar without the presence of Ecat was related to R2%-E0% with an increase of 14% compared to the reference (R0%-E0%). This increase was made by the bridging action of the RSF and the arrest of cracks from further openings. However, in the case of 3% of RSF in samples without Ecat, it was observed that the ultimate flexural capacity was only 6% increase compared to the reference. At this point, the authors did not study about the corresponding deflection behavior and post-cracking behavior and postpone it for future.

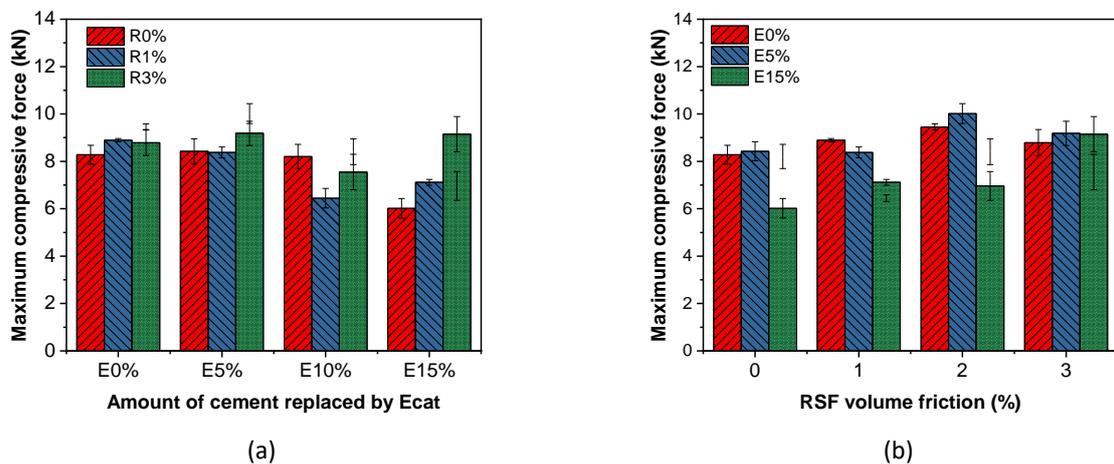
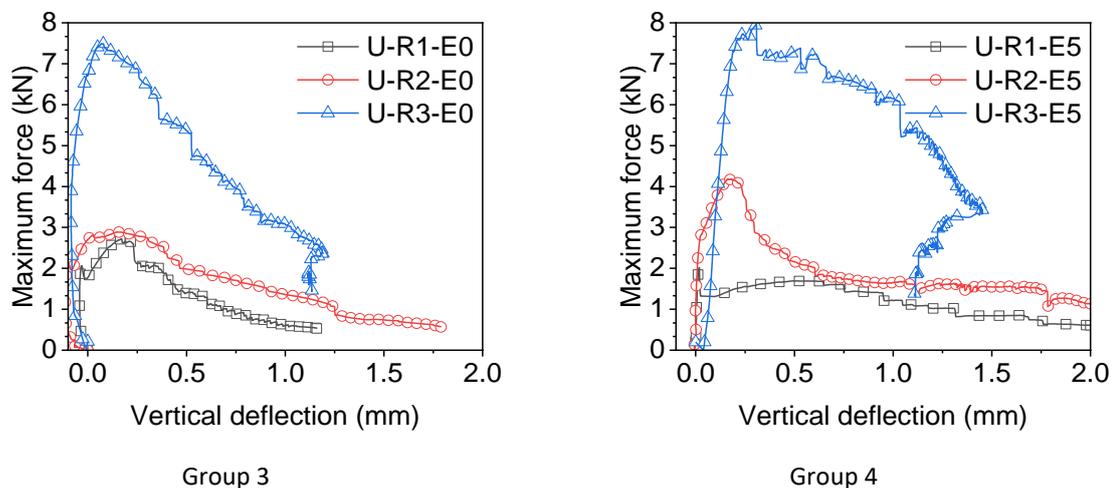


Figure 5. Maximum flexural force at seven days: (a) effects of RSF in different Ecat content, (b) effects of Ecat in different RSF content

Fig. 5b shows that, in the case of unreinforced mortars (RSF=0%) replacing cement with Ecat until 10%, it had no influence in decreasing the flexural stiffness of the tested specimens. It was observed that in the case of 15% replacement of cement with Ecat and using 3% RSF the flexural load increased compared to other mixes. This can be explained by the fact that the interface between the past aggregates and the fiber matrix was increased because of better performance of Ecat.

The results of the three point bending test of the notched samples are presented in Fig. 6. Fig. 6a shows that in the case of the mortars reinforced with 1% RSF (U-R1-E0), there was a linear increase in load with an increase in deflection until the maximum load value of 2.21 kN and vertical deflection of 0.03 mm. At this stage, a sudden degradation of the stiffness of the tested specimens occurred due to the creation of microcracks. The load decreased by approximately 28% (1.58 kN); however, the presence of RSF in the cement matrix prevented further propagation of the cracks. The bridging property of the fibers led to the hardening of the sample, which resulted in it withstand a load of 2.26 kN (with a deflection of 0.30 mm). Subsequently, the weakening of the sample was observed. There was a reduction in load and an increase in vertical deflection due to the appearance of cracks. It was concluded that the addition of 1% of the RSF was insufficient to increase the flexural strength of the beams and only resulted in the change of the failure mode from the brittle failure mode to the ductile one.

In the case of mortars with the addition of 2% and 3% fibers (U-R2-E0 and U-R3-E0, respectively), their behavior can be described as linear elastic, hardening, and softening. These three behaviors were more distinctive for the U-R3-E0. In the case of adding 3% of the RSF, the tested beams showed linear-elastic behavior until they obtained a load value of 2.5 kN and a vertical deflection of 0.15 mm. After this point, the occurrence of microcracks in the cementitious matrix resulted in a nonlinear response of the specimens, as well as their hardening behavior. After the load was increased to 5.95 kN, and the vertical deflection was equal to 0.37 mm, microcracks propagated along the cross section of the tested beams. At this point, the microcracks led to the loss of stiffness of the specimens. Stiffness was lost due to the pulling of the fibers from the cement matrix, which meant that the sample was weakened. When comparing the results for different amounts of RSF, the highest ductility and energy absorption were detected for specimens reinforced with 3% RSF.



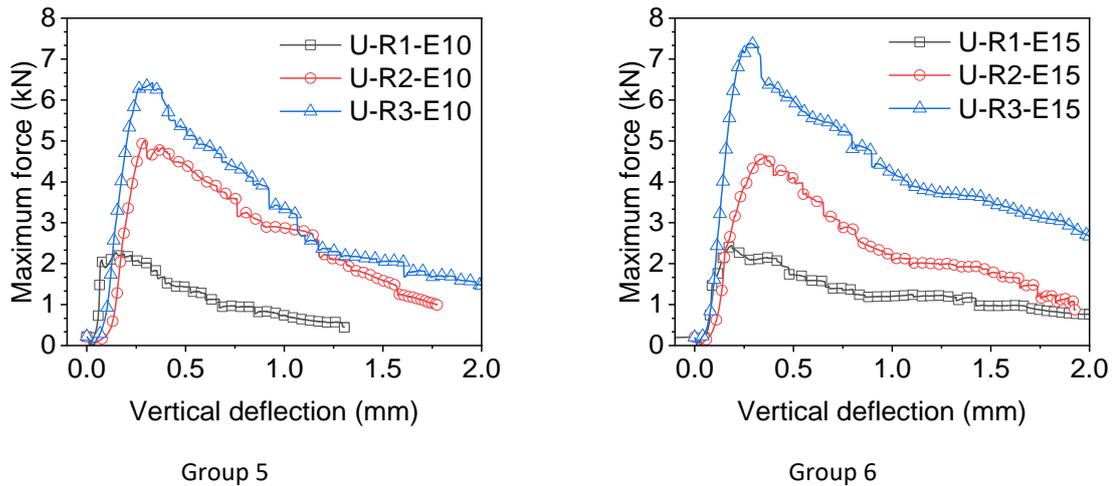


Figure 6. Maximum force versus vertical deflection in different groups of developed mortars

3. Conclusions

1- The assessment of the fresh phase of mortar showed that the addition of fiber created a high cohesive force between the mortar matrix and the RSF. Consequently, the workability decreased with increasing content of fibers.

2- Utilizing 1% and 3% of fiber dose resulted in a decrease in fallout after nearly 3% and 22% compared to the mortar without fibers. However, the three proposed mixes comply with the relative drop flow diameter criteria proposed for self-compacting mortars.

3- Beneficial effects of RSF inclusion in increasing compressive strength 7 days and 28 days were observed. Compared to mortar without any RSF, 1% and 3% inclusion of RSF resulted in increases for 18% and 24% for 7 days mortar and 16% and 22% for 28 days mortars, respectively. A similar improvement was observed for the tested beams under flexural loading conditions. It was noticed that the inclusion of RSF for 1% and 3% resulted in an increase in flexural strength for mortars of 1%, 2.5% for mortars of 7 days, and 7% and 8% for mortars of 28 days, respectively.

4- Regarding the notched beam specimens under flexural loading, it was concluded that using 1% RSF for reinforcing UHPSCM can improve the brittle behavior due to ductile failure while being not affected by the flexural strength of mortar. Significant improvement in tensile flexural behaviour was observed in the case of 3% RSF by the hardening and post-crack responses of the specimens.

5- Remarkable influences of the use of RSF were observed in modifying the failure modes of the specimens under compression and flexural loading. It was observed that, in the case of unreinforced mortar, the specimens explosively failed under compressive loading without any awareness of the inclined propagation of cracks with 45° that propagate from the bottom and top sides to the center specimens. In the case of both reinforced mortars with 1% and 3% fiber, the appropriate interaction between RSF and matrix prevented it to occur during the abrupt failure and changed to the ductile failure by gradual propagation of micro cracks along the specimens. Similar failure modes were noticed in the case of samples under flexural loading from the abrupt failure mode in the case of unreinforced mortar to the ductile one in the case of reinforced mortars.

6- Based on the significant advantages of the proposed mortars, the possibility of using these mortars for different purposes, such as retrofitting and / or flexural element without stirrups, can be recommended for future research lines.

4. Conflict of interest

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

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