

ANALYZING THE EFFECT OF MICRO RUBBER, MICRO SiO₂, AND NANO SiO₂ IN MICROCRACKS IN SELF-CONSOLIDATING CONCRETE (SEM OBSERVATION)

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ABSTRACT

The present study is an attempt to analyze the effect of micro rubber waste in self-consolidating concrete (SCC) and to compare the concrete containing SCC with conventional additives such as micro SiO₂ and nano SiO₂. The use of rubber waste can be substantially important from the environmental point of view. Hence, concrete specimens containing 1, 3 and 5% micro rubber waste were made. Moreover, specimens containing 1, 3 and 5% nano SiO₂ and 4, 8 and 12% micro SiO₂ were prepared to compare their behaviour and microstructure with each other and with the witness specimens. The effect of the other parameters such as the specimen age and the w/c ratio on the microstructure of concrete containing rubber waste was also studied. Thereafter, the specimens were imaged using a scanning electron microscope (SEM) to observe and compare the microcracks in the concrete and secondary electron beam (SE) was used to obtain their images. The results of the microstructural consideration of different specimens showed that 1% of micro rubber waste can improve the behaviour of self-consolidating concrete, but the concrete microstructure strength and quality decline with an increase in its amount.

KEYWORDS

Self-consolidating concrete, Micro rubber waste, Microcrack, SEM

INTRODUCTION

Self-consolidating concrete (SCC) has been shown to achieve the fresh- and hardened-state requirements for modern concrete construction [1-4]. The American Concrete Institute (ACI) defines SCC as concrete that is highly flowable without segregation that can self-fill complicated or congested-reinforcing steel formwork with very little mechanical-forced consolidation [5].

The use of self-consolidating concrete is increasing due to its advantages including the lack of need for vibration and easy implementation. In the past decades, numerous researchers have extensively studied the behaviour of this type of concrete. Some researchers have tried to improve the behaviour of this type of concrete by adding additives. For instance, Makul [6] tried to analyze the behaviour of self-consolidating concrete by adding rice husk ash. The use of rice husk ash along with the presence of foundry sand waste increased the strength of self-consolidating concrete. The analysis of concrete microstructure has also garnered the attention of researchers in recent years. The microstructural condition of concrete reflects its technical properties such as compressive strength. The use of SEM images is one of the best methods of observing the microstructure of concrete. The images captured by this type of microscope can contribute to the highly precise examination of the details of concrete cracks and microstructure [7-10]. Safiuddin et al. [11] explored the mechanical properties and microstructure of carbon fiber reinforced self-

consolidating concrete. The important mechanical properties of this type of concrete including compressive strength, splitting tensile strength, modulus of rupture or flexural strength, and toughness were estimated. The addition of carbon fibers to self-consolidating concrete improved almost all of the aforementioned parameters. Afterward, the microstructure of the self-consolidating concrete specimens was examined using SEM images.

In this research, the overarching goal is to study the effect of using micro rubber waste powder in the production of self-consolidating concrete and analyze the behaviour of self-consolidating concrete containing micro rubber waste powder. The effect of using this material on the microstructure of self-consolidating concrete is also studied using SEM images. SE beams are used to capture the images and study the microstructure of the specimens. In addition, to compare the effects of the presence of micro rubber waste powder with conventional concrete additives such as micro SiO₂ and nano SiO₂, the self-consolidating concrete specimens were prepared with different percentages of these materials to compare their behaviour and microstructure conditions with the specimens containing micro rubber waste powder.

EXPERIMENTAL PROGRAM

Self-consolidating concrete is one of the most commonly used and beneficial types of concrete that does not require vibration for implementation and is easy to implement. The goal of this research is to study the effect of the presence of micro rubber waste in this type of concrete. This study is conducted based on the compressive strength of concrete as well as its microstructure. To analyze the behaviour and microstructure of self-consolidating concrete specimens containing micro SiO₂ waste and specimens containing micro SiO₂ and nano SiO₂, 15x15x15 cm cubic specimens were made. Besides, two specimen ages including 7 and 28 days were used to analyze the effect of improving the microstructure on time. Moreover, all of the self-consolidating concrete specimens were made with the 0.4 and 0.5 w/c ratios to study the effect of these parameters on the behaviour and microstructure of concrete. Type 2 cement produced by Sistan Cement Company was used to make the specimens. The chemical analysis of this cement product is presented in Table 1 and the results of its physical tests are presented in Table 2. The mix design of the witness self-consolidating concrete specimens is also shown in Table 3. Different additive percentages are used in different specimens.

Tab. 1- Cement chemical analysis (percent)

SiO ₂	Al ₂ O ₃	Fe ₂ O ₃	CaO	MgO	SO ₃	Na ₂ O	K ₂ O	L.O.I	I.R	C ₃ S	C ₂ S	C ₃ A	C ₄ AF	F.CaO
21.47	5.4	3.82	62.52	1.31	2.88	0.65	0.43	1.52	0.72	41.33	30.02	7.84	11.6	1.49

Tab. 2- Results of the physical tests on concrete

Blaine (cm ² /g)	Setting time (Min)		Autoclave Exp%	Comp. strength (kg/cm ²)			
	Initial	Final		2 Days	3 Days	7 Days	28 Days
3110	183	238	0.08	-	265	344	451

Tab. 3- Self-consolidating concrete mix design

Material	Quantity (kg/m ³)	Proportion
Cement	400	1
Fine aggregate	610	1.525
Coarse aggregate	1170	2.925
Water	200	0.5
Super plasticizer	10	2.5

Rubber segments were converted into rubber particles as shown in Figure 1 to obtain the micro rubber waste powder. Thereafter, they were turned into micro rubber waste powder using an ICAN ball mill, which is depicted in Figure 2. Figure 3 depicts the final micro rubber waste powder.



Fig. 1 – The initial rubber powder



Fig. 2- The ball mill used for the production of micro rubber



Fig. 3- Micro rubber waste powder

After making the specimens according to different mix designs, all the specimens containing micro SiO_2 , nano SiO_2 and micro rubber waste were cured in water until they ruptured. Afterward, all specimens were broken with a digital jack at the same speed under equal conditions and their compressive strength was recorded. The location of the storage of specimens and the digital jack are shown in Figures 4 and 5, respectively.

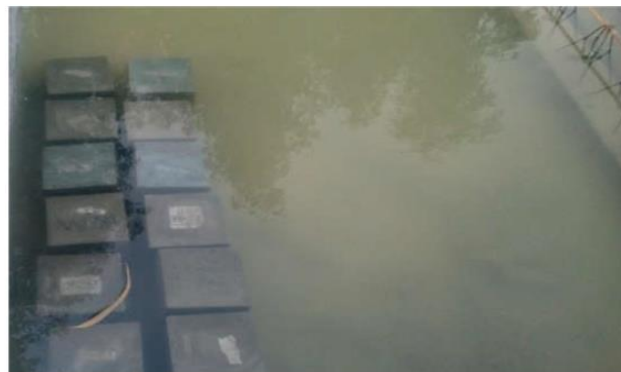


Fig. 4- Curing the specimens



Fig. 5- Testing the specimens' compressive strength

In this phase, the specimens were sent to the SEM laboratory of Sistan and Baluchestan University to study the microstructure of the specimens. The SEM device used is shown in Figure 6. Scanning electron microscopes basically operate different from optical microscopes. The microscope does not function based on the return of light. Rather, there is an electron gun at the top of the device that generates electrons at high voltage and shoots them at the specimen. The specimen surface is scanned by these electrons, and the electrons shot by the electron gun replace the electrons on the surface of the object. The electron shot by the surface of the object, which is known as a Secondary Electron (SE), hits the device detector and creates an image. Such a system is capable of providing highly accurate images of the specimen surface. The device is connected to a monitor that can display the SE images. The SE images are highly suitable for receiving the section roughness and showing the cracks and cavities.



Fig. 6 – The SEM device used for imaging

The use of the SEM device and production of appropriate images by this device depend on the qualification of the device operator because specialized operators can obtain better images than inexperienced operators. Before placing the specimens in the SEM, they have to be prepared. This process involves the dehydration of the specimens in vacuum, which is depicted in Figure 7. Thereafter, the specimens have to be covered in gold to be able to capture their images. The device that covers the specimens with gold is also shown in Figure 8. Figure 9 presents the dried specimens that are covered in gold and are ready to enter the SEM. The dimensions of these specimens are smaller than two centimeters. Basically, the specimens exceeding these dimensions cannot be placed in the device. The specimen preparation operations are highly precise and critical and take several hours. Precise preparation can significantly affect the results of imaging and the quality of the images.



Fig. 7 - Drying the specimens in vacuum



Fig. 8 – The machine that places gold covers on the specimens



Fig. 9 - Specimens ready for SEM imaging

Results and Discussion

The list of the specimens made and their compressive strength is presented in Table 4. As regards the names of the specimens in this table, the names of the witness specimens start with the letter W followed by their age and water to cement ratio. As for the other specimens, the first letter represents the additive (N = Nano SiO₂, M = Micro SiO₂ , MR = Micro Rubber) and the number after it is the percent of using the material. The numbers after S also show the age and the water to cement ratio, respectively.

Tab. 4- Specimens compressive strength

Specimen	Age (day)	W/C ratio	Nano SiO ₂ (kg/m ³)	Micro SiO ₂ (kg/m ³)	Micro Rubber (kg/m ³)	Compressive strength (MPa)
W74	7	0.4	————	————	————	24.5
W284	28	0.4	————	————	————	36.9
W75	7	0.5	————	————	————	21.3
W285	28	0.5	————	————	————	32.8
N1S74	7	0.4	1	————	————	21.4
N1S284	28	0.4	1	————	————	27.1
Specimen	Age (day)	W/C ratio	Nano SiO ₂ (kg/m ³)	Micro SiO ₂ (kg/m ³)	Micro Rubber (kg/m ³)	Compressive strength (MPa)
N3S74	7	0.4	3	————	————	28.0
N3S284	28	0.4	3	————	————	45.4
N5S74	7	0.4	5	————	————	25.3
N5S284	28	0.4	5	————	————	29.3
N1S75	7	0.5	1	————	————	17.9
N1S285	28	0.5	1	————	————	24.1
N3S75	7	0.5	3	————	————	24.3
N3S285	28	0.5	3	————	————	38.8

N5S75	7	0.5	5	—	—	19.5
N5S285	28	0.5	5	—	—	26.1
M4S74	7	0.4	—	4	—	43.0
M4S284	28	0.4	—	4	—	45.0
M8S74	7	0.4	—	8	—	50.6
M8S284	28	0.4	—	8	—	54.3
M12S74	7	0.4	—	12	—	48.3
M12S284	28	0.4	—	12	—	50.6
M4S75	7	0.5	—	4	—	34.1
M4S285	28	0.5	—	4	—	40.0
M8S75	7	0.5	—	8	—	36.3
M8S285	28	0.5	—	8	—	43.5
Specimen	Age (day)	W/C ratio	Nano SiO ₂ (kg/m ³)	Micro SiO ₂ (kg/m ³)	Micro Rubber (kg/m ³)	Compressive strength (MPa)
M12S75	7	0.5	—	12	—	29.6
M12S285	28	0.5	—	12	—	35.0
MR1S74	7	0.4	—	—	1	32.0
MR1S284	28	0.4	—	—	1	44.8
MR3S74	7	0.4	—	—	3	21.1
MR3S284	28	0.4	—	—	3	32.8
MR5S74	7	0.4	—	—	5	18.6
MR5S284	28	0.4	—	—	5	24.9

MR1S75	7	0.5	————	————	1	26.4
MR1S285	28	0.5	————	————	1	40.0
MR3S75	7	0.5	————	————	3	18.5
MR3S285	28	0.5	————	————	3	30.2
MR5S75	7	0.5	————	————	5	16
MR5S285	28	0.5	————	————	5	22.3

The diagram in Figure 10 shows the condition of the 7-day specimens with the 0.4 water to cement ratio. As seen, the specimens containing micro SiO₂ yielded the best results. Seemingly, at the early ages, the addition of 8 percent micro SiO₂ results in the highest strength level. Furthermore, the addition of a small amount of micro rubber, i.e. approximately 1 to 3 weight percent of cement, does not considerably affect the specimens' strength and their strength remains almost on the same level as the witness specimens. However, the specimen strength decreases with an increase in the micro rubber amount.

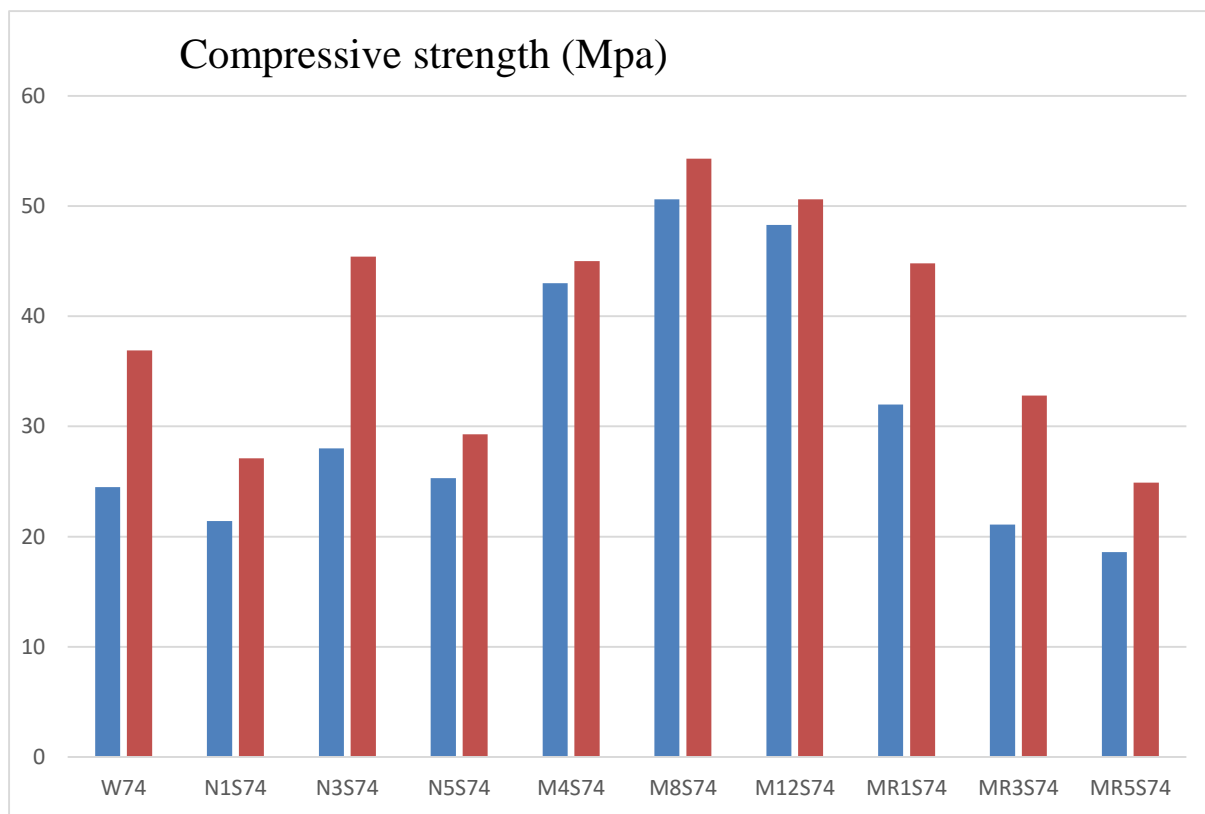


Fig. 10- The compressive strength of the specimens with the 0.4 water to cement ratio, the orange columns represent the same specimen at the age of 28 days.

Another finding is the rapid increase in the strength of the specimens containing micro SiO_2 , which gained a large percent of their compressive strength at the age of 7 days. However, in the specimens containing micro rubber, there is a considerable difference between the 7-day and 28-day strength levels similar to the witness specimen.

A comparison of the microstructures of some of these specimens explains their macroscopic behaviour. In Figure 11, the microstructure of specimen W74 is presented. As seen, the microstructure of this specimen is completely disintegrated and porous with numerous cavities. Figure 12 shows the microstructure of the specimen containing micro SiO_2 , which is more integrated and uniform with a stronger structure. This image clearly mirrors the effect of micro SiO_2 particles on the concrete integration.

Figure 14 indicates the compressive strength of the specimens with water to cement ratio of 0.5 at the ages of 7 and 28 days. Figures 15 and 16 also show the microstructure of the W75 and M8S74 specimens, respectively. As seen, the structure of the specimen containing micro SiO_2 is denser and more uniform, hence its higher strength. There are many more pores in the witness specimen. A comparison of both of these specimens with their similar specimens with a water to cement ratio of 0.4 reveals their weakness as well as their higher number of pores and lower strength. Considering the microstructure of the specimens containing micro rubber, which is shown in Figures 13 and 17, these microstructures are in a different condition. Although the porosity of the specimens containing micro rubber is lower and they have adequate integration, the mechanism of mixing with rubber is different in these specimens and the structure of CHs is also evidently different.

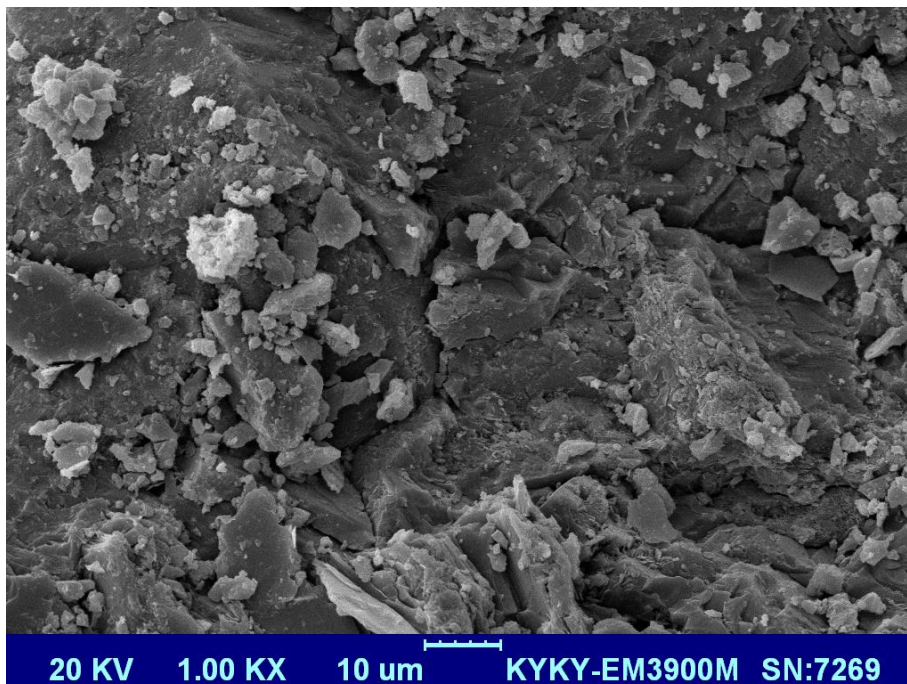


Fig. 11- The microstructure of the W74 specimen

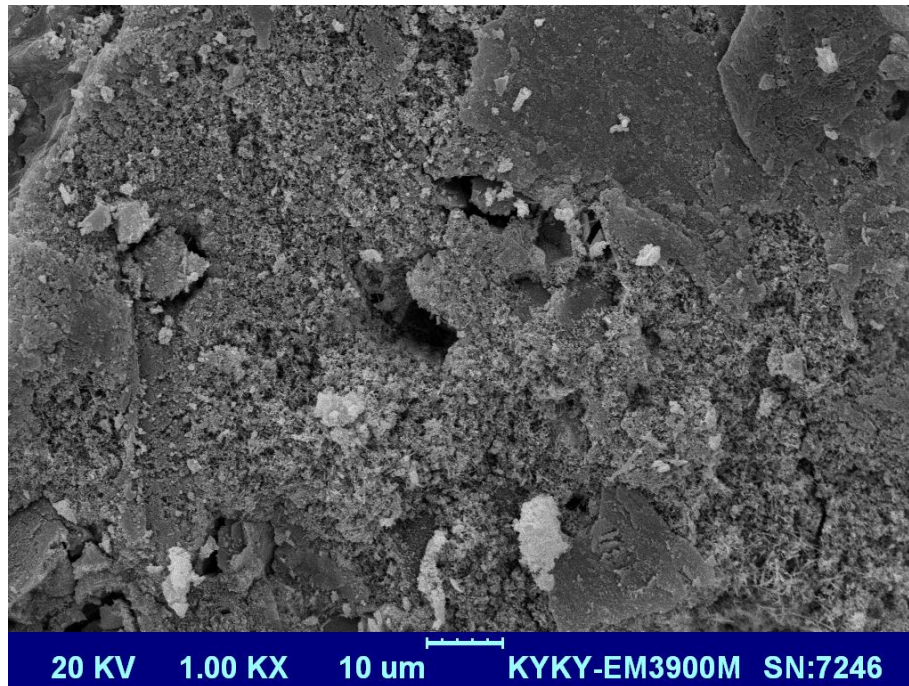


Fig. 12 - The microstructure of the M8S74 specimen

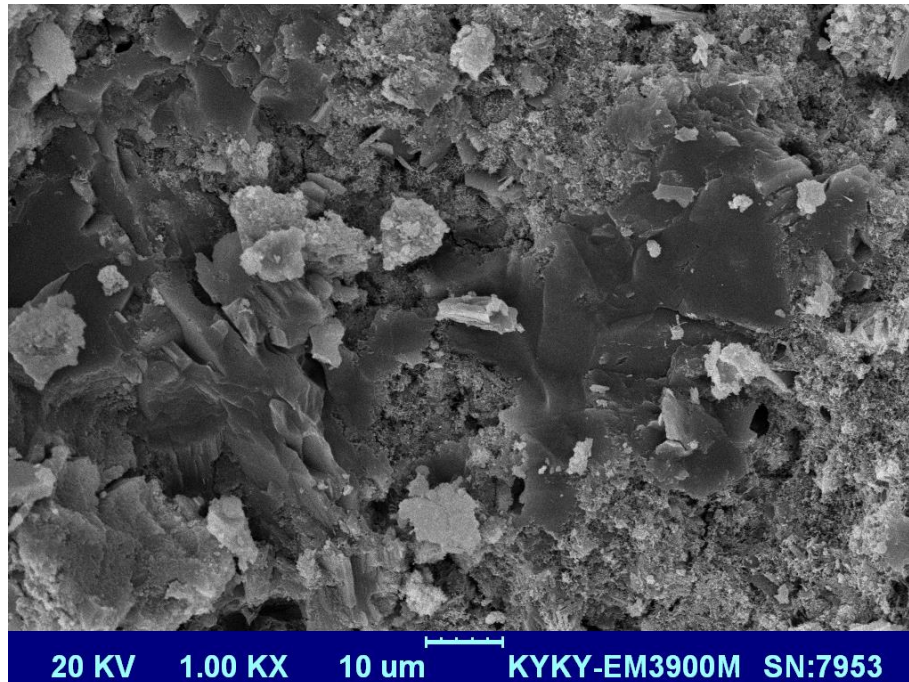


Fig. 13- The microstructure of the MR5S74 specimen

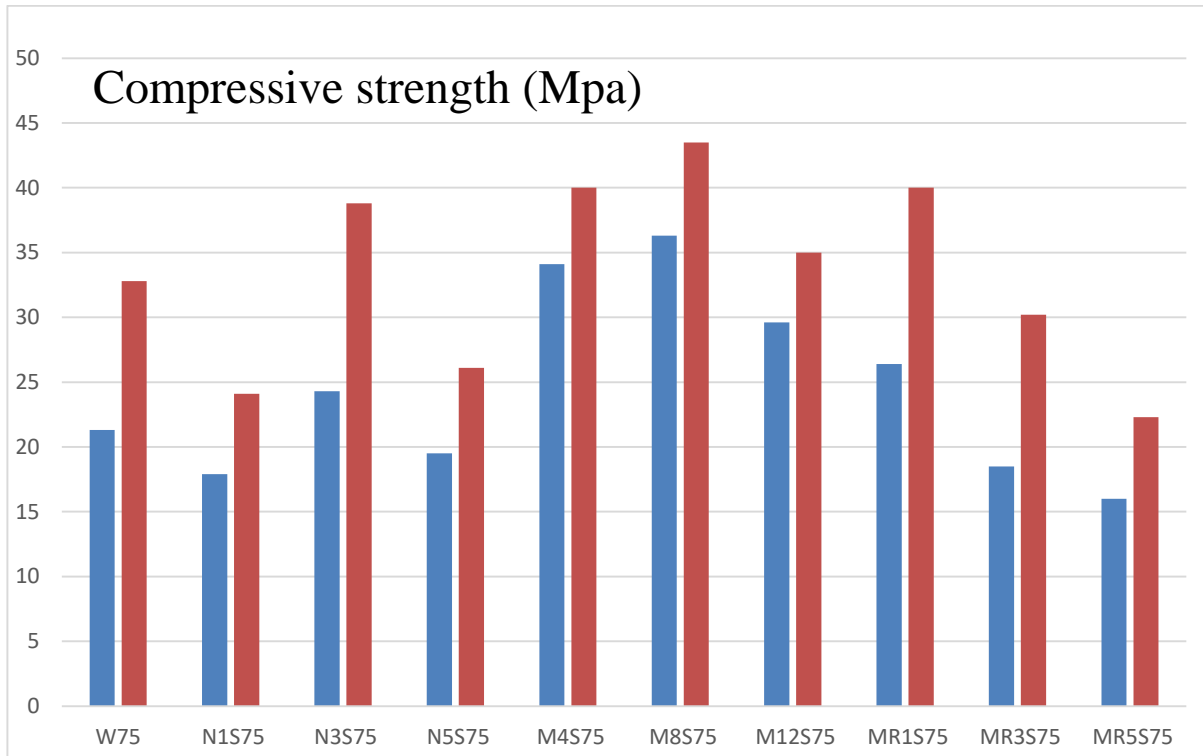


Fig. 14 – The compressive strength of the specimens with a water to cement ratio of 0.5, the orange columns show the same specimen at the age of 28 days.

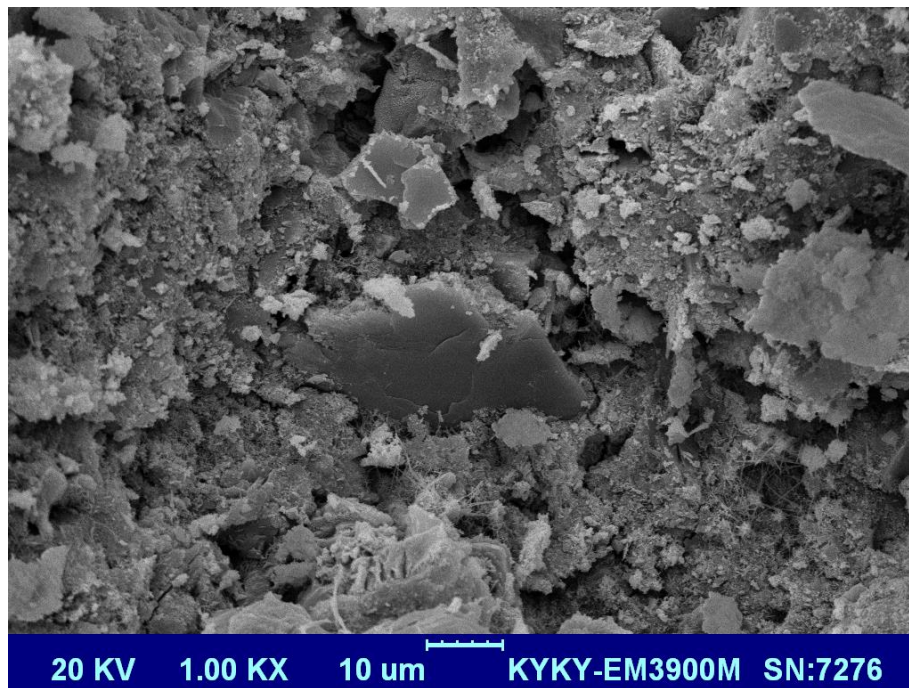


Fig. 15- The microstructure of the W75 specimen

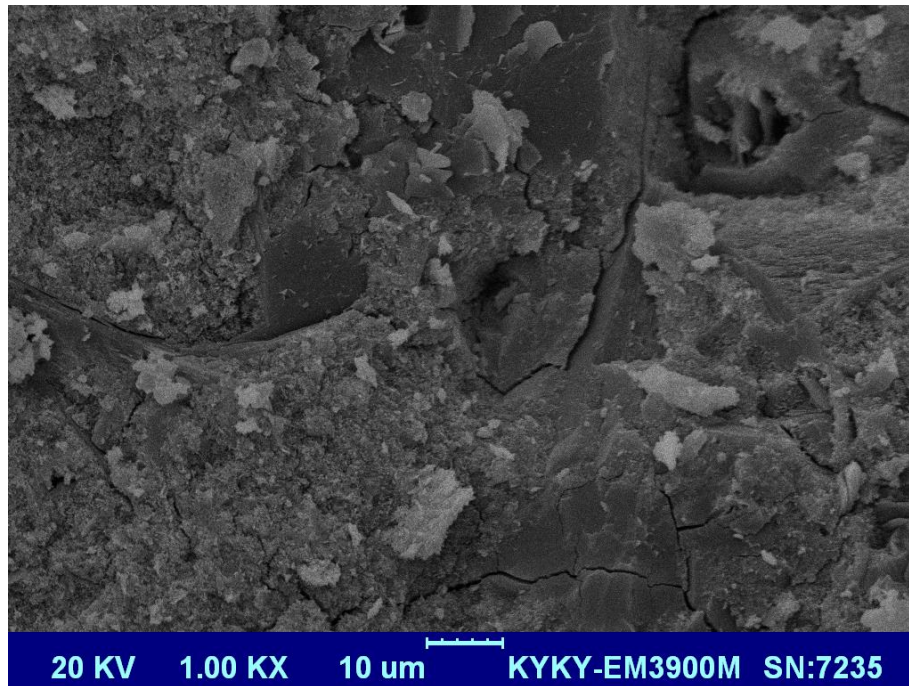


Fig. 16 – The microstructure of the M4S75 specimen

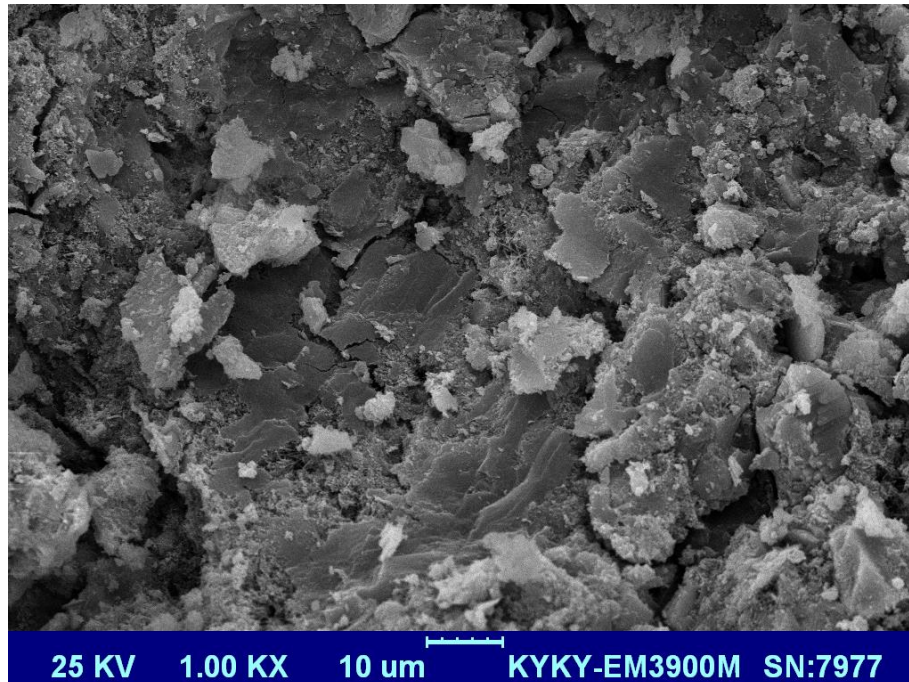


Fig. 17 – The microstructure of the MR5S75 specimen

CONCLUSIONS

- The addition of 1 percent of micro rubber waste can increase compressive strength by approximately 20% as compared to the witness specimen. If the amount of micro rubber increases to 3 and 5%, the compressive strength of the self-consolidating concrete specimen decreases as compared to the witness specimen.
- In all specimens, the uniformity of the microstructures increases with a decrease in the water to cement ratio and an increase in the strength and the microstructure of the specimens containing micro SiO₂ is still better and more integrated than the witness specimen containing micro rubber. Seemingly, micro SiO₂ can hold different parts of concrete together like an adhesive and create a more integrated specimen.
- As the age of the specimens increases to 28 days, the specimen containing 1% of micro rubber waste still has a higher strength than the witness specimen. In addition, even the strength of the specimen containing 5% of micro rubber waste at this age is approximately equal to the strength of the witness specimen, which indicates the approximate amount of micro rubber waste that can be used in concrete.
- In the microstructure of specimens with a water to cement ratio of 0.5, there are cavities both in the witness specimen and the specimen containing micro rubber cavities. These cavities are relatively large and cause the porosity of concrete. However, after adding micro SiO₂, these cavities merge and technically fade away.
- Using micro rubber waste powder in SCC mixtures can be a considerable environmental protection. In fact, the natural materials of SCC can substitute by waste one. Furthermore, using low percent of rubber waste powder (1 percent) not only can improve SCC compressive strength, but also can reduce the cost of SCC providing. So low amounts of micro rubber waste powder can be used commonly in SCC mixtures.

REFERENCES

- [1] H. Okamura, M. Ouchi. "Self-consolidating concrete, Development, present use and future" 1st International RILEM Symposium on Self-Consolidating Concrete (1999).
- [2] H. Okamura, M. Ouchi. "Self-consolidating concrete" J. Adv. Concr. Technol., 1 (2003), pp. 5-15
- [3] H. Okamura, M. Ouchi. "Application of self-consolidating concrete in Japan" 3rd International RILEM Symposium on Self-Consolidating Concrete (2003).
- [4] M. Ouchi. "Self-compactability of fresh concrete" 1st International RILEM Symposium on Design, Performance and Use of Self-Consolidating Concrete (2005).
- [5] American Concrete Institute ACI 237R-07 Self-consolidating concrete ACI Manual of Concrete Practice, Part 1, Farmington Hills, Michigan (2007).
- [6] N. Makul. "Combined use of untreated-waste rice husk ash and foundry sand waste in high-performance self-consolidating concrete". Results in Materials. 1 (2019) 10014.
- [7] Adili, E; Sohrabi, M. R; Nehi, H. M "Prediction of microcracks in concrete using fuzzy systems" journal of intelligent & fuzzy system. Vol 27. No 3. pp 1161-1168, 2014.
- [8] Diamond, S. and Huang, J. "The ITZ in concrete- a different view based on image analysis and SEM observation" Cement and Concrete Composites. 23 (2001) 179-188.
- [9] Feiteira, J; Tsangouri, E; Gruyaert, E; Lors, C; Louis, G and Debelie, N "Monitoring crack movement in polymer-based self-healing concrete through digital image correlation, acoustic emission analysis and SEM in-situ loading" *Materials & Design*, Vol 115, 5 February 2017, Pages 238-246.
- [10] A. Santamaría , A. Orbe, M.M. Losañez, M. Skaf, V. Ortega-Lopez, Javier J. González. "Self-compacting concrete incorporating electric arc-furnace steelmaking slag as aggregate". *Materials and Design* 115 (2017) 179–193.
- [11] Md. Safiuddin, M. Yakhlaf and K.A Soudki. "Key mechanical properties and microstructure of carbon fibre reinforced self-consolidating concrete". *Construction and Building Materials*. Vol 164. 2018. PP 477-488.