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Review article

The Effect of Elevated Temperature on Engineered Cementitious Composite Microstructural Behavior: An Overview

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ABSTRACT

This paper discusses the quantitative bibliographic data derived from scientific publications on Engineered Cementitious Composites (ECC) subjected to elevated temperature, the influence of elevated temperature on the mechanical properties, particularly the compressive strength and microstructure behavior of Engineered Cementitious Composites (ECC) mixtures based on the review of previous pieces of literature. Systematic literature reviews were employed as the methodology in this study. The age of related publications selected to be reviewed was limited to publications for the past ten years, 2010 to December 2020. It was found from available research that exposure of the ECC specimen at the elevated temperature starting from 200°C significantly reduced the compressive strength when the temperature increases, melting of fiber and increase of porosity causes the dramatically increase micro-cracks.

Keywords: Cracks, Engineered Cementitious Composites (ECC), elevated temperature, fiber, microstructure, scanning electron microscope (SEM)

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INTRODUCTION

Engineered Cementitious Composite (ECC) is a special type of High-Performance Fiber Reinforced Cementitious Composites, distinct for tensile strain-hardening behavior and tensile ductility compared to the quasi-brittle design of conventional concrete

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and fiber-reinforced concrete (Huang & Zhang, 2014; Khan et al., 2021). ECC has demonstrated excellent uniaxial tensile strain capacity of 3–5% (about 300–500 times higher than conventional concrete, which is 0.01%) and tight crack width of about 40 μm (Li, 2003; Ma et al., 2015). ECC incorporates industrial waste has also been studied for more greener and sustainable construction materials (Booya et al., 2020; Chen et al., 2013; Lee et al., 2019). The use of ECC has expanded dramatically over the last decade and has been used in a number of systems, such as high-rise buildings (Yang et al., 2021), bridges (Zhang et al., 2021), tunnels (Huang et al., 2021), highways (Guan et al., 2021) and other infrastructures. ECC is suggested in strengthening beam-to-column connections that are recurrently exposed to cyclic loading, a continuous and repeated loading, such as fluctuating stresses, strains, and forces, seismic actions (Lee et al., 2018; Qudah & Maalej, 2014). The ECC-concrete beams with ECC application at the tension region, when subjected to flexural load, displayed a substantial improvement in flexural strength and ductility, proven under both the experimental and theoretical aspects (Yuan et al., 2020; Zhang et al., 2006).

The study of rheological and mechanical properties of the ECC especially dealing with the risk of exposure to elevated temperatures has recently attracted attention. Table 1 shows the previous studies on ECCs subjected to elevated temperature, with different raw materials applications, geometry specifications, experimental testing, and heating parameters. It is found from Table 1 that common raw materials in ECCs are cement, mineral admixtures (i.e., silica fume, limestone powder, fly ash), sand (i.e., river, sand), fibers (i.e., PVA, PE), and superplasticizer. Previous studies investigated strength performances of ECC specimens under room temperature and temperatures at a range of 50°C to 1200°C. The ECC specimens were subjected to a heating rate of 4.4°C/min to 23.5°C with heating exposure for 1–2 hours. High temperature can cause changes in the physical and chemical properties of the ECC, which further affect the mechanical properties and microstructural behavior of the material. The effect of the elevated temperature on mechanical properties, particularly compressive strength and microstructural behavior, such as deterioration of fiber and microcracking that reduce the durability of the ECC, was seen clearly (Şahmaran et al., 2011). It is, therefore, necessary to fully understand the effects of high temperature on ECC so that the applications of ECC could be more effective and sustainable.

This paper presents an overview of the effects of elevated temperature on the microstructural behaviour of ECC. The systematic and integrative reviews were used as the methodology in this study. This study focuses on the selected related research articles limited to recent ten-years publications. This paper consists of four sections. Section 2 presents the methodology of the literature review. Section 3 presents the results of the bibliometric analysis of the topic. Section 4 discusses the effects of elevated temperature on the compressive strength, deterioration of fibers, and microcracking behavior of ECC. Finally, Section 5 presents the conclusions.

Table 1
Previous studies on ECCs subjected to high temperatures

Reference	Raw Materials	Sample size	Experimental testing	Targeted Temperature	Heating Rate	Heat Exposure Time, hour(h)
Bhat et al. (2014)	Type I Ordinary Portland Cement (OPC), class F fly ash (FA), polyvinyl alcohol (PVA) fiber, polycarboxylate-based high range water reducer (HRWR)	229×76.2×12.7mm	Uniaxial tensile test, Uniaxial compressive test	100°C, 200°C, 300°C, 400°C, 600°C	23.5°C/min	2 h
Mohammed et al. (2019)	OPC, nano-silica (NS), class F FA, PVA fiber, washed river sand (average size of 450 µm), HRWR	50mm cubes and dog-bone-shaped	Uniaxial tensile test, Uniaxial compressive test	100°C, 200°C, 300°C, 400°C	15°C/min	1 h
Luo et al. (2019)	OPC, Limestone Powder, Silica Fume (SF), Polyethylene (PE) fiber	40×40×160mm and 40×40×40mm	Uniaxial tensile and compressive test, Flexural test	20°C, 50°C, 100°C, 140°C, 200°C	6.6°C/min	2 h
Abdullah et al. (2013)	Class F FA, alkaline activator	50mm cubes	Uniaxial compressive test	400°C, 600°C, 800°C	4.4°C/min	1 h
Shang and Lu (2014)	Type I OPC, Class F FA, sand, water, PVA fibers, a polycarboxylic ether type HRWR, and hydroxypropyl methylcellulose	Dog-bone-shape	Uniaxial compressive test, Mass Loss	200°C, 400°C, 600°C, 800°C	13.3°C/min	1 h
Şahmaran et al. (2011)	Class F FA, PVA fiber, polycarboxylate-based HRWR	50mm cubes and 200×75×13mm	Uniaxial compressive test, Fire Resistance	200°C, 400°C, 600°C, 800°C	15°C/min	1 h
Wang et al. (2020)	OPC, river sand, PVA fibers	100mm cubes	Uniaxial compressive test	200°C, 400°C, 600°C, 800°C, 1200°C	20°C/min	1 h

METHODOS

A systematic literature review approach was employed to overview the microstructural behavior of ECCs after being subjected to elevated temperatures. This approach has been broadly used in many research works and can directly provide important findings from the existing field of knowledge (Ren et al., 2020). In addition to a systematic and comprehensive search to find all previous studies relevant to the research topic, this approach includes explicit criteria that can include or exclude studies to ensure the quality search of the literature. The review protocol for this study is illustrated in Figure 1.

Generally, the protocol consists of two main phases. In the first phase, the data for the bibliographic of the study of microstructure analysis on ECC subjected to elevated temperature was done from a complete retrieval from online databases in Scopus. Scopus is easily accessible, available in the organization subscriptions as well as it is notably with good coverage of publications at sufficiently high quality. The selections were then filtered to obtain the actual trend of the research. The trend of publication year and research work allocation were the data aimed for the retrieval. Computerized databases were used as an approach to obtain this literature because this approach is more efficient and effective.

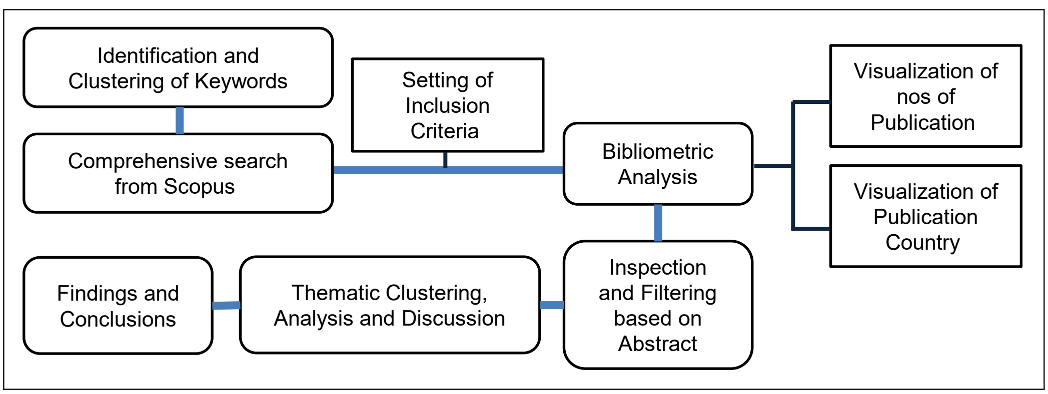

Figure 1. Review protocol

Table 2
Results of literature retrieval and selection

Steps	Descriptions	Records
Logical Statement in Advance Search	"ENGINEERED CEMENTITIOUS COMPOSITES” AND “ECC” AND ("ELEVATED TEMPERATURE" OR "HIGH TEMPERATURE") AND ("MICROSTRUCTURAL" OR "MICROSTRUCTURE" OR "MICRO STRUCTURAL" OR "MICRO STRUCTURE") AND ("scanned electron microscope" OR "electron microscope" OR "XRD" OR "SEM" OR "EDS")	Valid records (first-round filter): 90
Inclusion criteria	Publication years (2010 to Dec 2020), Type of publication: Article, Keywords, subject area: engineering and material science.	Final records (second-round filter): 52

The search started with all the literature based on the topics that related to the research's title. Then, the advanced search function in Scopus online collection database was used to search the related articles. Finally, keywords were used, such as Engineered Cementitious Composites, elevated temperature, microstructural, scanning electron microscope, and electron microscope. The actual coding for the advance search in Scopus is shown in Table 2.

The procedure followed the setting of inclusion criteria. These criteria include the publication years, publication types, refining keywords, and subject matters. Particularly, publication years were limited to 2010 to December 2020. The document types were confined to only research and review articles. Other types of literature, such as the book, conference paper, and book chapter, were not considered. Subject matters, such as engineering and materials science were included, and refining keywords, such as microstructure, scanning electron microscopy, engineered cementitious composite (ECC), elevated temperature, fire resistance, microcracks, microstructural analysis, micropores, x-ray diffraction, fire exposures, matrix microstructure, microstructural characteristics and microcracking were included. Figure 2 shows the number of articles after applying the inclusion criteria.

The entire abstract from 52 articles recorded from the second-round filter was clustered according to the themes. Next, the themes were identified to answer the research questions, such as (1) What are the effects of the temperature on the compressive strength of ECC, and (2) What are the effects of elevated temperature on microstructural behavior of ECC? Finally, the articles that fit the themes of the study were chosen for further analysis, data discussion, and evaluation.

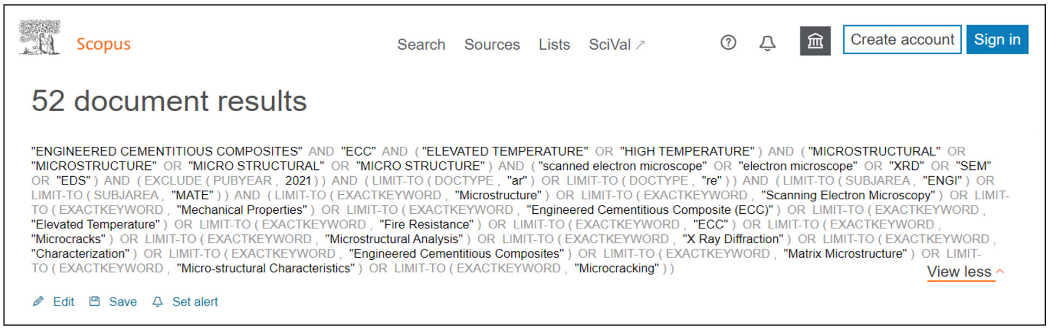

Figure 2. Logical statement and inclusion criteria (Image from Scopus online database)

RESULTS AND DISCUSSION

Bibliometric Analysis

Figure 3 shows the publications for the last ten years for the topics as filtered in Figure 2. Research related to the microstructural behavior of ECC after exposure to elevated temperature has gotten the attention of researchers in recent years. A significant increasing trend of publications can be seen from 2017 to 2020. The highest number of publications (16 nos) was recorded in 2020.

Figure 4 shows the statistical distribution of data number of publications per country for the last ten years. China has actively published articles and showed interest in the research regarding the exposure of ECC to elevated temperature (25 nos of publications), followed by the United States, recorded as 12 publications in years 2010-2020. On the other hand, very few works were done on this study in the countries of the Association of Southeast Asian Nations (ASEAN); only two publications were detected in Malaysia and Singapore, respectively.

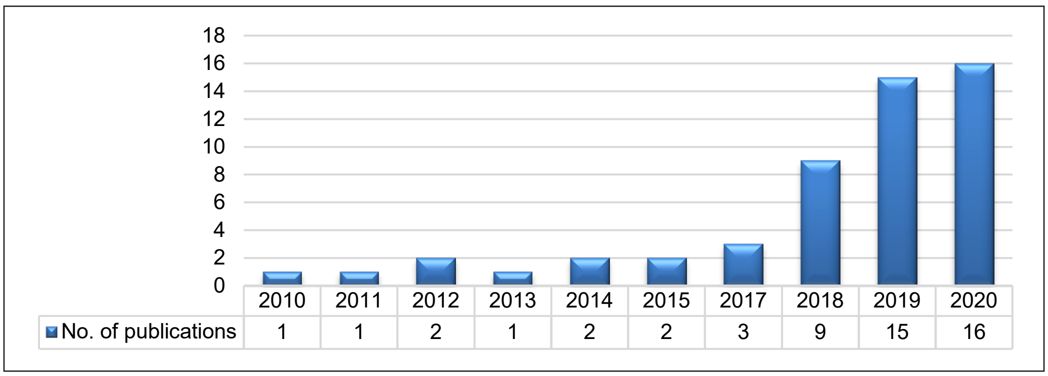

Figure 3. Numbers of publications per year (2010-2020)

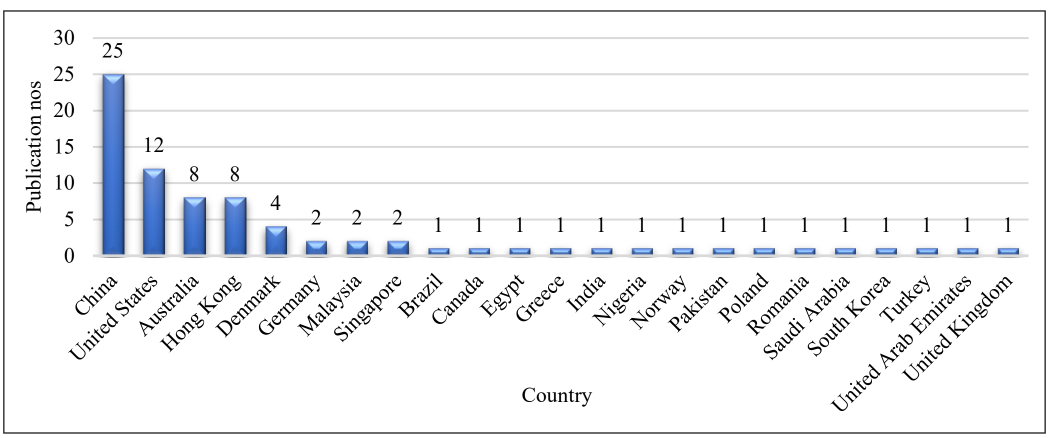

Figure 4. Statistical distribution of data number of publications per country

Effect of Temperature on Compressive Strength

Table 3 shows the compressive strength of ECCs after exposure to different temperatures. A decreasing trend in the compressive strength is observed when ECCs exposed to elevated temperatures starting from 200^{o}C. The decrement of the compressive strength is obvious

Table 3
Compressive strength of ECCs after exposure to different temperatures

Reference	Types of mineral admixture (MA/C)	Types of fine aggregate	Types of Fiber	Compressive strength after exposure to different temperatures							
				50°C	100°C	140°C	200°C	400°C	600°C	800 °C	1200°C
(Şahmaran et al., 2011)	FA (1.2) for ECC1	110 μm SS	PVA 2% v.f.	-	-	-	-14%	-15%	-47%	-66%	-
(Şahmaran et al., 2011)	FA (2.2) for ECC2	110 μm SS	PVA 2% v.f	-	-	-	-22%	-23%	-49%	-69%	-
(Shang & Lu, 2014)	FA (0.5)	Sand (size not specified)	PVA 2% v.f	-	-	-	+ 1%	-32%	- 62%	- 77%	-
(Luo et al., 2019)	LP (0.3), SF (0.25), GGBS (1.25)	100 μm SS	PE 2% v.f	+ 20%	+ 15%	- 8.3%	-50.5%	-	-	-	-
(Du et al., 2018)			PVA 2% v.f	-	-	-	-5.2%	-18.5%	-	-	-
(Yu et al., 2015)	FA	Sand	PVA 2% v.f	-	-	-	+3.6%	-11%	-32%	-74%	
(Aslani & Wang, 2019)	FA, SF	150 μm SS	PVA 1.75%	-	+30%	-	-	-26%	-52%	-70%	
(Wang et al., 2020)	-	River sand 2mm	PVA 2% v.f	-	-	-	-18.1%	-18.4%	-47.2%	-67%	-89.3

Note: + indicates an increase in compressive strength, - indicates a decrease in compressive strength
MA, FA, SF, SS denote mineral admixture, Fly ash, Silica Fume, Silica sand, respectively
v.f. denotes volume fraction (assume density of PVA fiber =1290kg/m^3, density PE fiber = 970kg/m^3)

after 400°C. Şahmaran et al. (2011) found a greater decrement in the compressive strength for ECC with higher fly ash volume at 200°C–400°C, however no significant effect of fly ash volume in the reduction at 600°C–800°C. According to Şahmaran et al. (2011), decreasing in compressive strength when exposed to 200°C is due to the hydration of fly ash particles, whereas at temperature 400°C is due to porosity increase and weight loss. ECC specimens demonstrated a great loss in compressive strength after being exposed at temperatures 600°C to 800°C. A greater reduction in compressive strength, especially at temperature 400°C to 800°C, was reported by Shang and Lu (2014) in their ECC specimens with lower fly ash content. When the ECC specimens were subjected to temperature 1200°C, the decrement up to 89% compared to the one at room temperature was noticed.

Luo et al. (2019) found enhancement in compressive strength when the ECC specimen exposure to a temperature lesser than 100°C. However, decreases steadily started from 50°C to 200°C. The higher temperature increased the pore size and porosity of the specimen. The drastic change in the compressive strength was due to visible minor cracking and increase of porosity (Luo et al., 2019; Şahmaran et al., 2011; Shang & Lu, 2014).

Heating duration can affect the residual mechanical properties of the specimen (Yu et al., 2015). For example, after being exposed for one hour at a temperature of 200°C, the compressive strength increased 3.6%. After two hours of exposer at the same temperature, the compressive strength of the specimen increased 11.5%. The changes in the mechanical properties of the specimen at this temperature may be due to the reinforced hardened cement paste (HCP) during the evaporation of free water, leading to greater Van der Waal forces as the cement gel layers pass closer to each other. The same goes for the study by Aslani and Wang (2019), whereby the compressive strength increases 30% after being exposed to 100°C. The decreasing of the compressive strength at temperature 400 might be due to small microcracks appearing and a drastic decrease of the compressive strength after exposer up to 600, maybe because this temperature level was a critical temperature for strength loss in ECC specimens.

Exposure of ECCs to high temperatures tends to experience moisture loss, and this scenario causes the increment of the initial porosity of ECCs. The increasing porosity resulting from high temperature coarsens the pore size and subsequently gives a remarkably negative effect to the compressive strength (Luo et al., 2019; Şahmaran et al., 2011; Shang & Lu, 2014). Melting of fiber in ECC at high temperature is the reason for the great number of pores and high porosity of the matrix. Figure 5 shows the cumulative pore volume of the ECC specimens after being exposed to the targeted temperature.

Most of the studies mentioned above investigated the strong performances of ECCs at exposure temperature 200°C and onwards. It is supported by Luo et al. (2019), where there are no significant changes in the strength before exposure to a temperature below 200°C. Although many have provided strength performance data at a temperature range from 200°C-800°C, ECCs investigated in these studies mainly incorporated PVA fiber at

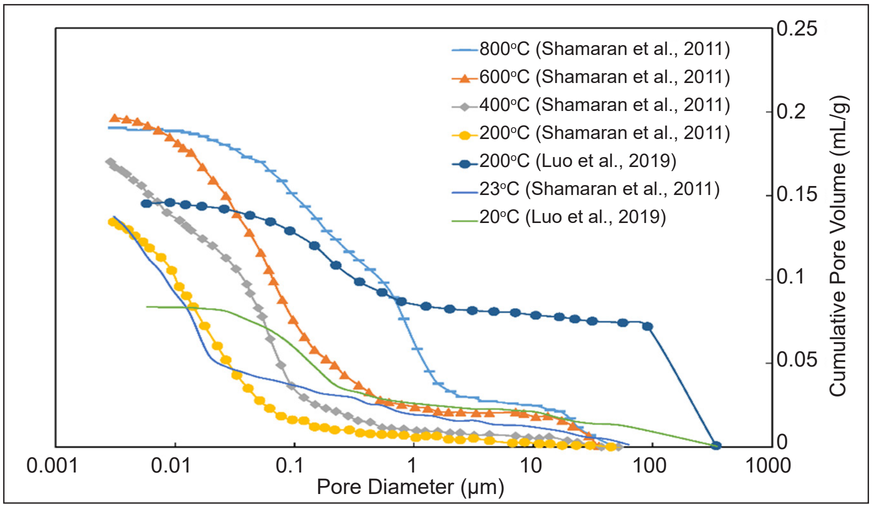

Figure 5. Cumulative pore volume of the sample before and after exposure to elevated temperature by (a) (Şahmaran et al., 2011), and (b) (Luo et al., 2019)

2% volume faction. Findings on this topic for other fibers (i.e., steel, PE fibers) and their percentages are very limited. Additionally, incorporating the mineral admixtures in these studies is in wide options (i.e., fly ash, silica fume, slag). These findings hardly conclude that they provide a real understanding of the behavior of the ECCs when exposed to elevated temperatures.

Microcracking and Fibre Deterioration

Table 4 presents the effect of different temperature levels on the deterioration of fiber and the microstructural behavior of ECC. The heating regime is explained based on four main temperature ranges, namely 0°C–200°C, 201°C–400°C, 401°C–600°C, and 601°C–800°C. The melting point of PE fibers in ECC is observed at 150°C, causing the increase of the porosity and the average pore size of the ECC specimen (Luo et al., 2019). On the other hand, the temperature effect on the microstructure of the fiber at 140°C caused the unrecoverable deformation, which affected the mechanical properties of the specimen. On the one hand, PVA fibers started to melt, and micropores began to appear at a temperature of 200°C (Bhat et al., 2014).

At temperature 400°C, the PVA fibers have melted completely and created micropores and interconnected pores in the ECC microstructure (Bhat et al., 2014; Mohammed et al., 2019; Şahmaran et al., 2011; Shang & Lu, 2014). More micropores were observed in the specimen that was exposed to a temperature level of 600°C. The average pore diameter size of the ECC specimen exposed to a temperature of 600°C rapidly changes caused by the totally melted fibers and water evaporation. The magnification of SEM by Bhat et al. (2014) was chosen to illustrate the effect of temperature on the PVA fiber (Figure 6).

For instance, microstructure did not experience any changes at temperature 20°C (Figure 6a). Fiber started to melt, and micropores appeared at 200°C (Figure 6b) while the fiber completely melted, and more micropores appeared at 400°C to 800°C (Figures 6c and 6d). Similar findings were reported by Wang et al. (2020), and three mechanisms were claimed to play roles: the effects of thermal expansion, volume expansion due to chemical reactions, and coarsening of pore structure.

The crystalline calcium hydroxide (CH) started to disappear after the specimen was exposed to a temperature of 100°C (Mohammed et al., 2019). After exposure at a temperature of 200°C, tiny micro-cracks started to visible. The number and size of the micro cracks at this temperature also increase (Shang & Lu, 2014). Conversely, Şahmaran et al. (2011) reported no significant changes in the microstructure, and micro-cracks were not apparent at a temperature of 200°C.

Specimens exposed to the temperature of 300°C and 400°C became porous. The more micro-crack appeared at this temperature level due to the melted PVA fibers, dehydration, and degradation of the cementitious material (Mohammed et al., 2019). However, microcrack only started to be visible at 400°C in ECCs (Abdullah et al., 2013; Şahmaran et al., 2011).

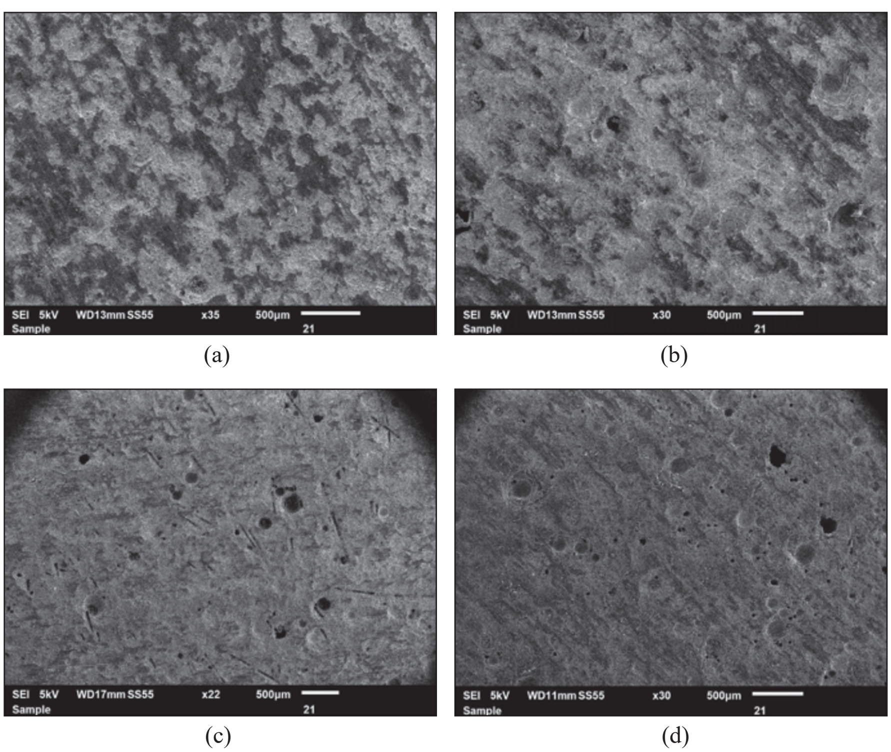

Figure 6. Magnification scanning electron microscopy of ECC subjected to elevated temperature: (a) 20°C; (b) 200°C; (c) 400°C; and (d) 600°C (Bhat et al., 2014)

At a temperature of 600°C and 800°C, rough heterogeneous with a larger proportion of pores appear. The visibility of rough pores at higher temperatures is due to the sintering process (Abdullah et al., 2013). At a higher a temperature of 600°C and 800°C, a wider micro-cracks appearance and a larger pore proportion due to the rate of water evaporation of the sample is high and the sintering process also high (Abdullah et al., 2013). The microstructure of the specimen exposed to a temperature of 800°C started to lose the crystalline strictures characteristic (Şahmaran et al., 2011; Shang & Lu, 2014).

Porosity increased after the specimen was exposed to temperature 200°C due to the pore water in the specimen starting to lose (Du et al., 2018). The melting of the PVA fiber at temperature 300°C created the amount of the pores to become larger. As the temperature increased, the amount of melted fiber also increased. PVA fiber completely melted after exposer at a temperature of 400°C. The surface of the fiber was moderately ruined; however, but the PVA fiber was observed not melting after exposure to a temperature of 200 (Yu et al., 2015). The fiber was completely melted at temperature 400°C, creating more pores and microcracks increased. The specimen loses its crystalline characteristic after being exposed to a temperature up to 800 (Aslani & Wang, 2019; Yu et al., 2015).

CONCLUSION

This paper presents quantitative bibliographic data derived from scientific publications and an overview of Engineered Cementitious Composites subjected to elevated temperature. There have been very limited studies on this topic for the past ten years. However, an increasing trend in the number of publications over the years reveals the importance and relevancy of the topic in the future, particularly in the construction industry.

The exposure of the ECC specimens to elevated temperatures can influence the mechanical properties of concrete, particularly in compressive strength. Most of the studies showed a decrement in compressive strength after being exposed to a temperature at 200°C onwards. It is found from previous researchers' findings that the decrement in compressive strength is due to the increase of pores, which are mainly caused by the evaporation of water and melting of fibers. The melting point of Polyethylene (PE) fibers at 150°C and Polyvinyl alcohol (PVA) fibers in between 201°C–400°C were reported. After the fibers started to melt, the microstructure behavior of the specimen changed, such as crystalline characteristics, pores, and microcracking was observed.

The residual properties of the specimen drop moderately after the ECC specimen is exposed to elevated temperature. Apparent microcracking and explosive spalling occurred in specimens subjected to elevated temperature compared to the unheated samples. When the temperature increases, micro-crack size dramatically increases and results in plastic behavior and unrecoverable shapes of fibers.

Table 4
Microcracking behavior

Author	0°C-200°C	201°C-400°C	401°C-600°C	601°C-800°C
Bhat et al. (2014)	-	Micro pores started to appear due to fiber beginning to melt	Fiber has melted and created more micropores. Micro-cracks appear as a "spider web" pattern.	-
Mohammed et al. (2019)	After the exposure at temperature 100°C, the crystalline calcium hydroxide (CH) started to disappear. At a temperature of 200°C, the microstructure started to become pores.	PVA fibers started to melt and cause the microstructural to become more pores and increasing micro-cracks.	-	-
Luo et al. (2019)	At temperature 140°C, PE fibers started to damage. Due to the melting of PE fibers at temperature 200°C, the average pore size increased compared to the specimen at room temperature (104nm to 203.09nm)	-	-	-
Abdullah et al. (2013)	-	Microcracks started to appear at a temperature of 400°C with an average width of microcracks between 0.1m to 0.2m.	After being exposed at a temperature up to 600°C, wider microcracks were noticeable.	At a temperature of 800°C, the average microcracks size became wider.
Shang and Lu (2014)	PVA fiber did not melt. A very small amount of microcracks start to appear.	PVA fibers completely melt at temperature 400°C. The number of microcracks was increased.	-	Specimen lose the characteristic crystalline structure after being exposed at temperature 800°C. The average pore diameter also increases.

Table 4 *(continue)*

Author	0°C-200°C	201°C-400°C	401°C-600°C	601°C-800°C
Şahmaran et al. (2011)	Microstructure did not experience many changes. Microcrack did not make apparent after being exposed at 200°C.	PVA fibers completely melt at temperature 400°C. Cracking and pores started to visible.	At temperatures over 600°C, the changes in average pore diameter appeared more rapidly. The sample also started losing its crystalline characteristic.	Increasing microcracking after exposure over 800°C. The average pore diameter significantly increases.
Du et al. (2018)	Pore water of the specimen exposed to temperature 200°C was lost, and porosity was observed to be increased.	At a temperature of 300°C, the volume of PVA fibers decreased due to the fiber had reached the melting point. The number of pores increased.	At a temperature of 400°C, melting of the fibers increases the number of pores and causes more cracks to appear.	-
Yu et al. (2015)	At a temperature of 200°C, PVA fibers did not melt, but the surface of the fiber was moderately ruined.	Fibers completely melted at a temperature of 400°C and created pores. Microcracks increased.	-	The number of microcracks increased at temperature 800°C, and the specimen started to lose crystalline structure.
Aslani and Wang (2019)	Temperature below that 100, PVA fiber still undamaged.	The surface of the fiber started to damage at temperature 300, and pores and microcracks started to be visible.	At temperature 600, PVA fiber completely melted, and microstructural became pores.	Specimen lose the characteristic crystalline structure after being exposed at a temperature of 900°C. The number of microcracks increased.
Wang et al. (2020)	Light grey color. No rupture of PVA fibers, but the diameter of fibers was reduced by 43%.	Whitish grey color. Melting of PVA and fibers in longilineal needle-like channels were observed.	Yellowish grey color. The dents are left by fiber and increase of average pore diameter and total pore volume. Hairline cracks started to be observed.	Light brown color. Channels left by PVA fibers were channels were gradually filled with newly produced.

Based on the previous research findings towards engineered cementitious composite subjected to elevated temperature, the paper makes recommendations on a few future perspectives of research with the aims to improve the current knowledge of findings:

- Advanced engineered cementitious composite. In recent years, significant numbers of smart, green, and innovative materials or techniques, such as hollow glass microspheres (Aslani et al., 2021), ceramic polishing brick powder (Xiong et al., 2021), microbially induced calcium carbonate precipitation using Aerobic non-ureolytic bacteria (Justo-Reinoso et al., 2021) have been utilized into ECC to enhance the material sustainability in the construction industry. However, studies on the effects of these materials on the strength and durability performances of the ECC subjected to elevated temperature are very new research and need to be conducted.
- Advanced microstructural characterization techniques. Many advanced techniques were recently proposed in examining microstructural characteristics of a material, such as the machine-based learning method (Chan et al., 2020; Liu et al., 2021) and deep convolutional neural networks (Dong et al., 2020). New potential methods need to be explored to obtain a better understanding of the microstructural characteristics of the ECC after being exposed to elevated temperatures.

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