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Effects of nanomaterials on hydration reaction, microstructure and mechanical characteristics of cementitious nanocomposites: A review

Kim, Gwang Mok*

Abstract: Application of nanomaterials to cementitious composites has been attempted with the rapid development of nanotechnology since the 1990s. Various nanomaterials such as carbon nanotube, graphene, nano-SiO_2, nano-TiO_2, nano-Al_2O_3, nano-Clay, and nano-Magnetite have been applied to cementitious composites to improve the mechanical properties and the durability, and to impart a variety of functionality. In-depth information on the effect of nanomaterials on the hydration reaction, the microstructure, and the mechanical properties of cementitious nanocomposites is provided in the present study. Specifically, this paper mostly deals with the previous studies on the heat evolution characteristics of cementitious nanomaterials at an early age of curing, and the pore and the compressive strength characteristics of cementitious nanocomposites. Furthermore, the effect of nanomaterials on the cementitious nanocomposites was systematically discussed with the reviews.

Key Words: Cementitious nanocomposites, Microstructure, Hydration reaction, Compressive strength

1. Introduction

Portland cement is one of the most representative construction materials used for hundreds of years. This material consists of a calcium silicate system and reacts with water (Brown, 1999). This reaction is called hydration. The major phases of the cement due to hydration reaction is calcium silicate hydrates (C-S-H) that mainly contributes to the mechanical properties of cement pastes and concretes (Famy et al., 2003). In addition, various phases including ettringite ($Ca_6Al_2(SO_4)_3(OH)_{12} \cdot 26H_2O$), and portlandite ($Ca(OH)_2$) could be formed during hydration reaction (Famy et al., 2003).

The application of nanomaterials to cementitious composites has been attempted with the rapid development of nanotechnology since the 1990s. Various nanomaterials such as carbon nanotube (CNT), graphene, nano-SiO_2, nano-TiO_2, nano-Al_2O_3, nano-Clay and, nano-Magnetite have been applied to cementitious composites to improve the mechanical properties and the durability and to impart a variety of functionality such as piezoresistive sensing, heating, electromagnetic shielding, high strength, radiation shielding, self-cleaning properties (Chen et al., 2011; Pan et al., 2015; Jo et al., 2007; Chen et al., 2012; Barbhuiya et al., 2014; Morsy et al., 2010; Amin et al., 2013).

In-depth information on the effect of nanomaterials on the hydration reaction, the microstructure, and the mechanical properties of cementitious nanocomposites is provided in the present study. Specifically, this paper mostly deals with the previous studies on the heat evolution characteristics of cementitious nanomaterials at an early age of curing, and the pore and the compressive strength characteristics of cementitious nanocomposites. Furthermore, the effect of nanomaterials on the cementitious nanocomposites was systematically discussed with the reviews.

2. Hydration characteristics of cementitious nanocomposites at an early age of curing

Figure 1 shows the representative schematics on the heat evolution of cementitious materials during hydration (Shi and Day, 1995). The heat evolution characteristics of cementitious composites can be categorized with five zones (Zone 1: pre-induction period, Zone 2: induction period, Zone 3: acceleration period, Zone 4: deceleration period, Zone 5: diffusion period)

* 한국지질자원연구원 광물자원연구본부 선임연구원, 교신저자 (k.gm@kigam.re.kr).

and there are two characteristic peaks (Shi and Day, 1995). The occurrence of the first peak in the zone 1 is attributable to the formation of primary C-S-H phases, while the second peak is caused by the formation of secondary C-S-H or other hydrates (Huanhai et al., 1993). Despite the schematics can be slightly varied with the composition of cementitious materials, the characteristic peaks are effective means to compare the hydration characteristics of cementitious materials at an early age of curing. Thus, the effects of nanomaterials on the hydration characteristics of cementitious materials in the present study were investigated with the characteristic peaks.

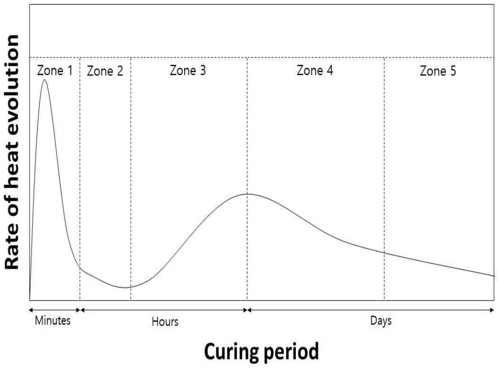

Figure 1. Representative schematics of heat evolution characteristics of cementitious materials during hydration (Shi and Day, 1995)

There are few available data with respect to the effect of nanomaterials on the occurrence of the first peak in heat evolution curves of cementitious nanocomposites. Li et al. (2017) added a graphene oxide of 0.2 % by the weight of cementitious materials and reported that the addition of graphene oxide reduced the occurrence time of the first peak in the heat evolution curves less than 42% compared to cementitious materials without graphene oxide. In addition, the rate of hydration heat at first peak increased more than 100% compared to that of cementitious materials without graphene oxide. Hou et al. (2017) added a graphene oxide of 0.16 wt% to cementitious composites and reported that the addition of graphene oxide reduced the occurrence time more than 10%. The rate of hydration heat at the first peak also increased more than 17%. That is, the addition of nanomaterials to cementitious nanocomposites in the previous studies reduced the occurrence time of the first peak and increased the rate of hydration heat. Kim et al. (2019) reported that the addition of nanomaterials could provide nucleation sites, thereby promoting the hydration reaction at an early age of curing. However, a further study is needed to deeply evaluate the effects of nanomaterials on the first peak in the rate of heat evolution curves.

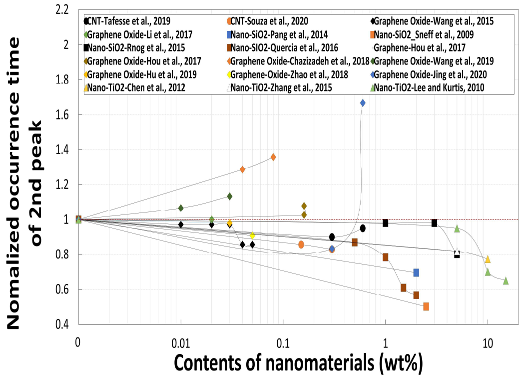

Figure 2. Effect of nanomaterials on the occurrence time of second peak in heat evolution curves of cementitious nanocomposites

Figure 2 shows the effect of nanomaterials on the occurrence time of the second peak in heat evolution curves of cementitious nanocomposites (Tafesse et al., 2019; Wang et al., 2015; Li et al., 2017; Souza et al., 2020; Pang et al., 2014; Senff et al., 2009; Rong et al., 2015; Quercia et al., 2016; Hou et al., 2017; Ghazizadeh et al., 2018; Wang et al., 2019; Hu et al., 2019; Zhao et al., 2018; Jing et al., 2020; Chen et al., 2012; Zhang et al., 2015; Lee et al., 2010). It should be noted that the normalized occurrence time of the second peak in this figure was calculated with the occurrence time of the second peak of cementitious materials without nanomaterials in each literature. The summarized results indicated that the addition of nanomaterials mostly reduced the occurrence time of the second peak compared to cementitious composites without nanomaterials. However, the addition of graphene oxide to cementitious composites in some literatures was ineffective to promote the occurrence of second peak (Wang et al., 2019; Hou et al., 2017; Jing et al., 2020). The clear difference between graphene (including graphene oxide) and other nanomaterials mentioned in the present study is morphology. Graphene oxide has a planar shape, while the other nanomaterials mentioned in the present study have spherical or fiber types (Cai et al., 2016). The morphological difference could affect the occurrence time of the second peak. That is, the graphene oxide could readily cover cementitious particles and inhibit the hydration reaction at an early age of curing (Kim et al., 2016a).

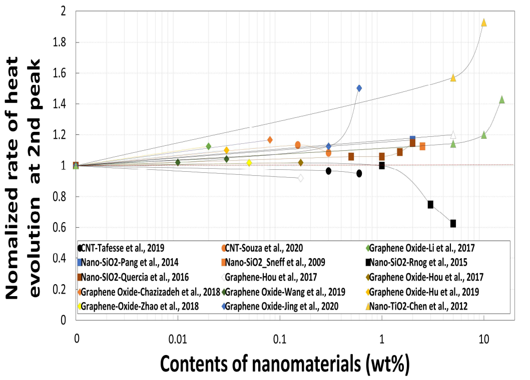

Figure 3. Effect of nanomaterials on the rate of heat evolution at second peak of cementitious nanocomposites

Figure 3 shows the effect of nanomaterials on the rate of the heat evolution at second peak of cementitious materials (Tafesse et al., 2019; Souza et al., 2020; Li et al., 2017; Pang et al., 2014; Senff et al., 2009; Rong et al., 2015; Quercia et al., 2016, Hou et al., 2017; Chazizadeh et al., 2018; Wang et al., 2019; Hu et al., 2019; Zhao et al., 2018; Jing et al., 2020; Chen et al., 2012; Zhang et al., 2015; Lee and Kurtis, 2010). The summarized results indicated that the rate of heat evolution at the second peak mostly increased when nanomaterials was added to cementitious composites. However, the rates of heat evolution at the second peak were reduced when the contents of CNT and nano-SiO_2 were more than 0.3 wt% and 1.0 wt%, respectively. It is well known that the hydration reaction of cementitious particles is initiated from the dissolution of reactive components from the particles (Kim et al., 2017). Kim et al. (2019) reported that the proper content of CNT added to cementitious nanocomposites to improve the mechanical properties was less than 0.2 wt% since an excessive addition of nanomaterials can lead to the covering cementitious particles, inhibiting the hydration reaction.

Furthermore, nanomaterials have strong Van der Waals attraction force (Kim et al., 2016a). The attraction force causes agglomeration of nanomaterials and thereby the dispersion state of nanomaterials could be poor (Kim et al., 2016a). The agglomerates of nanomaterials could entrap water to be used for hydration reactions (Kim et al., 2016a). That is, nanomaterials could significantly reduce the free water in the unhardened cementitious matrix (Kim et al., 2018). Overall, this phenomenon could reduce the rate of heat evolution at the second peak when a large amount of nanomaterials is added.

Meanwhile, the addition of nano-TiO_2 more than 5.0 wt% increased the rate of heat evolution at second peak (Lee and Kurtis, 2010; Chen et al., 2012; Zhang et al., 2015). The increase in the rate of heat evolution at the second peak was more than 10 % when nano-TiO_2 was added more than 5.0 wt%, while the increase in the rate of heat evolution was mostly less than 10 % when the other nanomaterials were added more than 1.0 wt%. In general, a proper content of nanomaterials to improve mechanical properties of cementitious nanocomposites is approximately less than 0.2 wt%. However, the content of

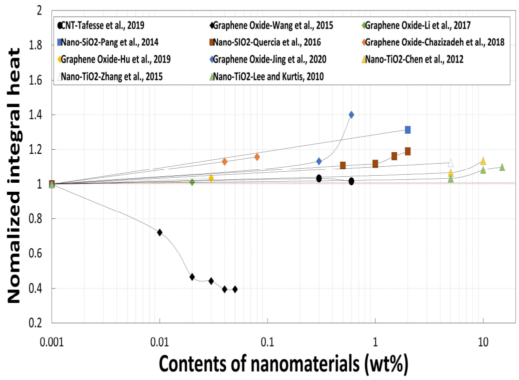

Figure 4. Effect of nanomaterials on integral heat of cementitious composites

nano-TiO2 in the previous study is generally higher than 5.0 wt%. Besides, the addition of nano-TiO_2 more than 5.0 wt% increased heat evolution at the second peak compared to other nanomaterials. The reason will be discussed later.

Figure 4 shows the effect of nanomaterials on the integral heat of cementitious materials (Tafesse et al., 2019; Wang et al., 2015; Li et al., 2017; Pang et al., 2014; Quercia et al., 2016; Chazizadeh et al., 2018; Hu et al., 2019; Jing et al., 2020; Chen et al., 2012; Zhang et al., 2015; Lee and Kurtis, 2010). The summarized results indicated that the addition of nanomaterials mostly increased the integral heat of cementitious nanocomposites. In contrast, Wang et al. reported that the addition of graphene oxide reduced the integral hydration heat (Wang et al., 2015). It was inferred in this study that the acceleration of hydration leading the occurrence of second peak when adding graphene oxide formed excessive hydrates on the surface of cementitious particles and thereby the dissolution of reactive components for hydration at the later stage was inbihited (Wang et al., 2015). This phenonena could result in the reduction of integral hydration heat.

In addition, for the nano-TiO_2, the increase in the integral heat was not significant compared to the other nanomaterials, despite the increase in the rate of the heat evolution at the second peak was significantly higher than the other nanomaterials. This was attributable that the acceleration of hydration reaction at the second peak when adding nano-TiO_2 formed a large amount of hydrates, which might reduce the reactive surface area of cementitious particles. That is, the hydrates, were possibly covered the cementitious particles, afterward inhibiting the hydration reaction (Kim et al., 2016b). Consequently, the addition of TiO_2 could reduce the integral heat after the occurrence of the second peak.

3. Microstructure of cementitious nanocomposites

Figure 5 shows the effect of nanomaterials on the total porosity of cementitious nanocomposites at 28 days of curing (Tafesse et a., 2019; Wang et al., 2015; Rong et al., 2015; Wang et al., 2019; Zhao et al., 2018; Chen et al., 2012; Liu et al., 2018; Nochaiya and Chaipanich, 2011; Kang et al., 2015; Du and Dai, 2015; Wang et al., 2016). The summarized results indicated that the addition of nanomaterials to cementitious nanocomposites reduced the total porosity. The total porosity in most cases was reduced in the range from 10 % to 40 %. The reduction in the total porosity of cementitious nanocomposites induced by the filling pores and the formation of hydrates (Kim et al., 2018). The gel pores in cementitous matrix are in the range of less than 10 nm, which is involved in the formation of hydrates (Dong et al., 2017). The capillary pores are in the range from 10 nm to 10 μm, which is the space after the evaporation of water in the cementitious matrix (Dong et al., 2017). In particular, a significant reduction in the total porosity in the case of Wang et al. 2015 was observed. It was reported in this study that the addition of graphene oxide to cementitious materials led the regular alignment of hydrates as well as filling pores and acceleration of hydration. The complex phenomena significantly reduced the total porosity (Wang et al., 2015). However, a further study is needed to deeply understand the exceptional effect of graphene oxide on the hydration reaction and microstructural characteristics of cementitious composites.

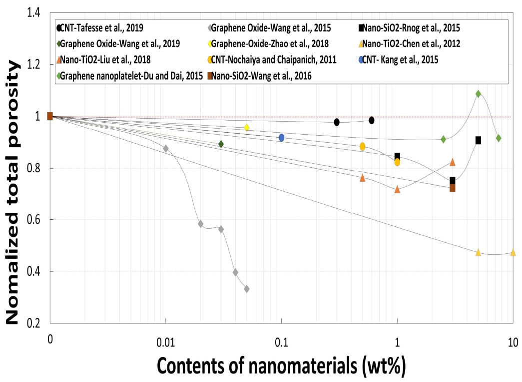

Figure 5. Effect of nanomaterials on total porosity of cementitious composites

Table 1 shows the relationship between normalized integral heat and normalized total porosity of cementitious nanocomposites. The summarized results indicated that the normalized integral heat was reduced or slightly increased as the contents of nanomaterials increased. In contrast, the total porosity was significantly reduced as the contents of nanomaterials increased, excluding the addition of CNT case. It can be inferred from the results indicated that the reduction in the total porosity of cementitious nanocomposites was mainly affected by filling the pores with nanomaterials.

Table 1. Relationship between normalized integral heat and normalized total porosity of cementitious nanocomposites

Nanomaterial	Content (wt%)	△H	△P	Ref.
CNT	0.3	1.03	0.97	Tafesse et al., (2019)
	0.6	1.01	0.98	
Graphene Oxide	0.01	0.72	0.87	Wang et al., (2015)
	0.02	0.48	0.58	
	0.03	0.44	0.56	
	0.04	0.39	0.39	
	0.05	0.39	0.33	
Nano-TiO_2	5.0	1.06	0.47	Chen et al., (2012)
	10.0	1.13	0.47	

△H: Normalized integral heat
△P: Normalized total porosity at 28 days of curing

In particular, for the nano-TiO_2, Chen et al., (2012) reported that the integral heat slightly increased when adding nano-TiO_2, while the total porosity was significantly reduced. In the previous study, the integral heat increased less than 15%, while the total porosity was reduced more than 50%. The results also indicated that most of nano-TiO_2 particles contributed to filling the pores and thereby reduced the total porosity. Nano-TiO_2 is not pozzolanic material. In contrast, pozzolanic materials such as nano-SiO_2 can contribute to the microstructural modification by the pozzolanic reaction due to the considerable surface activity (Qing et al., 2007). That is, the addition of nano-TiO_2 to cementitious nanocomposites mainly contributed to filling the capillary pores (pore size > 10 nm) (Dong et al., 2017).

However, a further study is needed to investigate the role of nanomaterials on the microstructural modification of cementitious nanocomposites, since the available data is few.

4. Mechanical properties of cementitious nanocomposites

Figure 6 shows the effect of nanomaterials on the compressive strength of cementitious nanocomposites at 28 days of curing (Tafesse et al., 2019; Wang et al., 2015; Rong et al., 2015; Wang et al., 2019; Zhao et al., 2018; Chen et al., 2012; Liu et al., 2018; Kang et al., 2015; Du and Dai, 2015; Wang et al., 2016; Li et al., 2017; Souza et al., 2020; Chazizadeh et al., 2018; Hu et al., 2019; JHing et al., 2020; Zhang et al., 2015; Wang et al., 2016). The summarized results indicated that the addition of nanomaterials to cementitious nanocomposites increased the compressive strength. The compressive strength of nanocomposites mostly increased from 5 % to 20 %. However, the compressive strength of cementitious composites in some cases was reduced. The reduction in the compressive strength of cementitious nanocomposites can be caused by several reasons. For example, the total porosity of cementitious nanocomposites can increase when excessive contents of nanomaterials are added, since the agglomerates of nanomaterials could contain water that is capable of forming voids in cementitious matrix (Li et al., 2017). Besides, the nanomaterials as themselves can cover the cementitious particles and thereby inhibit the hydration reaction (Kim et al., 2017). Similarly, the excessive promotion of nucleation due to the addition of nanomaterials at an early age of curing can form a large amount of hydrates

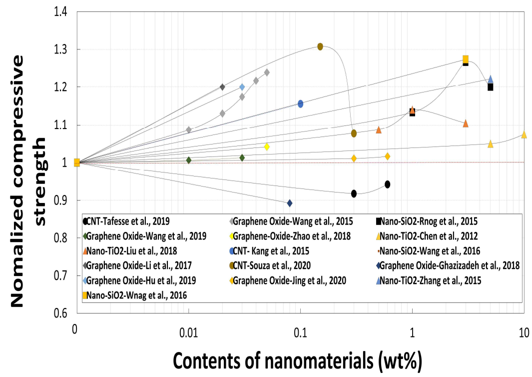

Figure 6. Effect of nanomaterials on compressive strength of cementitious composites at 28 days

surrounding cementitious particles, reducing the reactive surface area (Kim et al., 2018).

Table 2 shows the relationship between normalized total porosity and compressive strength at 28 days of curing. The summarized results indicated that the total porosity of cementitious nanocomposites at 28 days was mostly reduced when nanomaterials were added, and the compressive strength was also increased. In particular, the use of nano-SiO_2 was effective to improve the compressive strength of cementitious nanocomposites, despite the reduction in the total porosity of cementitious nanocomposites incorporating nano-SiO_2 was not significant compared that of the other nanocomposites. Nano-SiO_2 being a pozzolanic material could participate in the hydration reaction. The Si component could be directly consumed by the formation of C-S-H phases in cementitious nanocomposites (Khaloo et al., 2016;). That is, nano-SiO_2 particles could react in a cementitious matrix, improving the compressive strength.

Meanwhile, the increase in the compressive strength in some cases was reduced when the contents of nanomaterials increased, increasing the total porosity. As aforementioned, the phenomena was attributable that the agglomerates of nanomaterials entrapping water led to the formation of voids when excessive contents of nanomaterials was added. Consequently, the total porosity increased and thereby the increase in the compressive strength was slightly reduced.

The increase in the compressive strength in the previous studies was within 20 % despite the fractional reduction in the total porosity was in the range from 3 % to 70 %. It canbe inferred from the summarized results that there is a limit to the contribution of the densification by the incorporation of nanoparticles on the increase in the compressive strength of cementitious nanocomposites.

Table 2. Relationship between normalized total prositiy and normalized compressive strength of cementitious nanocomposites at 28 days

Nanomaterial	Content (wt%)	△P	△C	Ref.
CNT	0.3	0.97	0.91	Tafesse et al., (2019)
	0.6	0.98	0.98	
	0.1	0.91	1.15	Kang et al., (2015)
Nano-SiO_2	1	0.84	1.13	Rong et al., (2015)
	3	0.75	1.26	
	5	0.90	1.2	
	3	0.72	1.27	Wang et al., (2016)
Graphene Oxide	0.01	0.87	1.08	Wang et al., (2015)
	0.02	0.58	1.13	
	0.03	0.56	1.17	
	0.04	0.39	1.21	
	0.05	0.33	1.23	
	0.03	0.89	1.01	Wang et al., (2019)
	0.05	0.95	1.04	Zhao et al., (2018)
Nano-TiO_2	5.0	0.47	1.05	Chen et al., (2012)
	10.0	0.47	1.07	
	0.5	0.76	1.08	Liu et al., (2018)
	1.0	0.71	1.14	
	3	0.82	1.10	

△P: Normalized total porosity at 28 days
△C: Normalized compressive strength at 28 days

Table 3 shows the relationship between the normalized integral heat and the compressive strength at 28 days of curing. The summarized results indicated that the compressive strength of cementitious nanocomposites at 28 days mostly increased when the integral heat increased at an early age of curing. However, the compressive strength at 28 days in some cases was reduced, despite the integral heat at an early age of curing increased. It can be inferred from the results that the addition of nanomaterials negatively affect additional hydration and the growth of hydrates after initial hydration and. Besides, the compressive strength in other cases increased, despite the integral heat was reduced. That is, the densification induced by the addition of nanomaterials mainly contributed to improving the compressive strength of cementitious nanocomposites.

Table 3. Relationship between normalized integral heat at an early age of curing and normalized compressive strength of cementitious nanocomposites at 28 days

Nanomaterial	Content (wt%)	△H	△C	Ref.
CNT	0.3	1.03	0.91	Tafesse et al., (2019)
	0.6	1.01	0.98	
Graphene Oxide	0.01	0.72	1.08	Wang et al., (2015)
	0.02	0.48	1.13	
	0.03	0.44	1.17	
	0.04	0.39	1.21	
	0.05	0.39	1.23	
	0.08	1.15	0.89	Ghazizadeh et al., (2018)
	0.03	1.03	1.2	Hu et al., (2019)
	0.3	1.13	1.01	Jing et al., (2020)
	0.6	1.4	1.01	
	0.02	1.01	1.2	Li et a.l., (2017)
Nano-TiO_2	5.0	1.06	1.05	Chen et al., (2012)
	10.0	1.13	1.07	
	0.5	1.36	1.08	Liu et al., (2018)
	1.0	1.36	1.14	
	3.0	1.54	1.10	
	5.0	1.12	1.22	Zhang et al., (2015)

△H: Normalized integral heat at an early age of curing
△C: Normalized compressive strength at 28 days

5. Concluding remarks

The effects of nanomaterials on the hydration characteristics of cementitious materials at an early age of curing, the microstructural modification and the compressive strength of cementitious nanocomposites were investigated by reviewing previous studies. The main conclusions obtained in the present study are as follows.

(1) The addition of nanomaterials to cementitious nanocomposites promoted the occurrence of the first and the second characteristic peaks in the heat evolution curves. Furthermore, the use of nanomaterials mostly increased the rate of heat evolution at the second peak and the integral heat.

(2) The addition of nanomaterials mostly reduced the total porosity of cementitious nanocomposites. The reduction in the total porosity of cementitious nanocomposites was mainly attributed to the densification induced by filling pores with nanomaterials.

(3) The use of nanomaterials reduced total porosity and increased the compressive strength of cementitious nanocomposites. However, excessive use of nanomaterials increased the total porosity owing to its agglomeration and thereby reduced the compressive strength.

(4) The addition of nanomaterials mostly increased the integral heat at an early age of curing and the compressive strength of cementitious nanocomposites at 28 days, simultaneously.

(5) The shape and surface activity of nanomaterials possibly affected the hydration characteristics and compressive strength of cementitious nanocomposites

It can be concluded that the effects of nanomaterials on hydration characteristics at an early age of curing, microstructural modification and the compressive strength of cementitous nanocomposites was positive. However, the shape, the surface activity, contents and agglomeration characteristics of nanomaterials could negatively affect the properties.

감사의 글

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