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Influence of Fe_2O_3 Nanoparticles on the Characteristics of Waste Marble Powder Mixed Cement Mortars

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Abstract

Eco-friendly and durability of material has been the focuses of different researches; however, limited are considered both the nanomaterial and byproduct. Therefore, the aimed of this study is to investigate the influence of nano-Fe_2O_3 (NF) on various properties of cement mortar with marble powder (MP) as supplementary cementitious material (SCM). Fresh and hardened properties of each mix were examined. The microstructure of hydrated blended cement mortars was evaluated by scanning electron microscopy and X-ray energy-dispersive techniques. The effect was evaluated using flow table, compressive, split tensile, and durability tests. In general, the MP dosage mixes showed increase in water content due to high fineness and NF increased the workability and rate of hydration. The refined pores and enhanced C–S–H gel formation showing better strength with NF than MP. The mechanical properties result compared to control specimens showed that (MP + NF > NF > MP > Control). The optimal value MP and NF in cement mortar's strength and durability is 10% MP and 1% NA. The findings showed that the use of MP and NF in concrete technology as partial replacement of cement is effective and enhance the properties of cementitious composites.

Keywords Nano Fe_2O_3, Marble powder (MP), Microstructure, Porosity, Permeability

1 Introduction

Environment impact is critical issue that needs more investigation. Amid the causes to the impact construction industry has a significant role, which mainly resulted due to cement production that dramatically increasing every time. Among the billons of tons of cement production 0.8 tons per ton of carbon is released to the environment (Kumar & Prasad, 2019). This adverse effect needs to minimize(Ashish, 2019; ; Suman et al., 2021).Therefore, numerous researchers have been developing supplemental cementitious materials (SCMs), which can substitute cement (Dinakar et al., 2013; Amin et al., 2013; Maguesvari & Sundararajan, 2017; Sata et al., 2016; Sujjavanich et al., 2017; Zaetang et al., 2017), such as fly ash, ground granulated blast furnace slag, and silica fume(Lee et al., 2021; Lovecchio et al., 2020). Hence, finding new materials that replace cement is still required more research.

Cement replacement using byproduct is advantages in waste and pollution reduction and cost effective. Amongst marble powder is large and common byproduct dispose 25% of original marble in production and covers vast damping area that causes environmental pollution (Li et al., 2004). Incorporating marble powder in concrete improved workability and enabled the dispersion and compaction that helps to increase strength (Khan et al., 2021).The strength parameters are enhanced in a concrete contains MP due to its high reactive minerals, which can generate calcium silicate

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while reacting with calcium hydrate (Alyamac et al., 2016). The addition of marble powder in concrete mix can enhance compressive and flexural strength (Praveenkumar et al., 2019; Soliman, 2013) As a result, number of research has been done to explore the use of MP in concrete (Khan et al., 2021; Li et al., 2004).

Nanomaterial has unique structure and properties, such as high surface area, Young's modulus, strength, and electrical conductivity. As well as accelerates calcium silicate hydrate (C–S–H) gel formation with size ranged between 0.1 and 100nmthatcan improve other materials property and strength. This can apply in different industries including construction. Because of this typical behavior it mostly influence the chemical bonding at the interface and their small size leads to a larger surface area to volume, thus making them very reactive. In the field of construction material, such as cement, the use of nanoparticles is getting more attention in recent years (Ashraf et al., 2018; Feng et al., 2013; Kawashima et al., 2013; Kumar et al., 2021; Oltulu & Sahin, 2013). Due to the ultrafine particle size, the use of nanoparticles in concrete improved the different parameters (Shah et al., 2016). Iron oxide and other metal oxide nanoparticles are constantly being studied for their wide range of uses (Khoshakhlagh et al., 2012; Norhasri et al., 2017). Nano Fe_2O_3 particles play a role of nanofillers and enhance the susceptibility to water permeability of concrete (Jo et al., 2007; Nazari et al., 2010). The chloride permeability decreases by around 44% while adding nano Fe_2O_3 particles in cement composites (Nazari & Riahi, 2011) this indicated that the addition of NF particles increases the compressive strength of high strength concrete (HSC). The main failure of concrete is penetration of sulfate and chloride in to porous matrix due to poor compaction, dispersion, and hydration inside the cement structure (). Therefore, it is important to combine MP and NF to improve dispersion, hydration, and strength. Moreover, there are also limited comprehensive investigations available on the influence of nano Fe_2O_3 particles upon the characteristics of marble powder supplementary cementations materials mortar. Thus, the aim of this work is to identify the properties of modified cement mortar through marble powder (MP) and nano Fe_2O_3 particles (NF).

2 Materials and Methods

2.1 Materials

The material used in this study is a commercially obtained 43 grade OPC conforming IS: 8112 (IS 8112: Indian Standard, 2013) having initial and final setting times of 141 and 357 min, respectively. Natural river sand corresponding to grading zone-II with a specific gravity of 2.61, fineness modulus of 3.01, and bulk density of 1.48was used as fine aggregate conforming IS: 383 (IS, 1970). MP having particle sizes of 5–15 µm and NF having particle sizes of 20–25 nm were employed. Table 1 shows the chemical compositions of OPC and MP. Fig. 1a–c exhibits the XRD humps of MP, NF and OPC, respectively.

2.2 Mix Proportions

In this investigation, a total of 10 (triplicate) mix proportions, including control samples, were examined. For preliminary investigation, 1:3 cement mortars were made with W/B (water/binder) of 0.5 and were substituted in percentages of 5, 10, 15, and 20% by weight of the cement; meanwhile, NF also partially replaced with 0.5%, 1%, 1.5%, and 2% by weight of cement. The optimal dosage of MP and NF were determined using trial and error method and the same mix proportion was used for further investigation.

The fresh mortar specimens were prepared in such a way that first appropriate weighted dry constituents were placed in a mechanical mixer and allowed to mix for 1 min. And then, appropriate amount of NF and water are mixed together then allowed to mix with dry constituents for 4 min to ensure a homogenous blend. According to ASTM C311-16 (ASTM, 2016),the freshly prepared composites are put into cylindrical as well as cubical molds and compacted by utilizing tamping rods. The mixes were allowed permitted to cure for 24 h at room temperature on a level surface before being removed from the molds, and 28 day curing was performed.

2.3 Test Methods

2.3.1 Workability

Immediately after mixing all the components, the workability of the freshly mixed mortar was evaluated using a common flow table test according to ASTM1437-16 (ASTM, 2015a). The mixture was kept in a mold and compacted using a tamper to create a 25 mm layer. Following that, the second layer was filled all the way to the top of the mold and compacted in the same way.

Table 1 Composition of OPC and marble powder

Composition (%)	SiO_2	Al_2O_3	Fe_2O_3	CaO	MgO	SO_3	LOI
OPC	21.0	5.4	4.6	63.0	0.7	2.9	2.5
MP	18.43	–	–	67.79	13.78	–	–

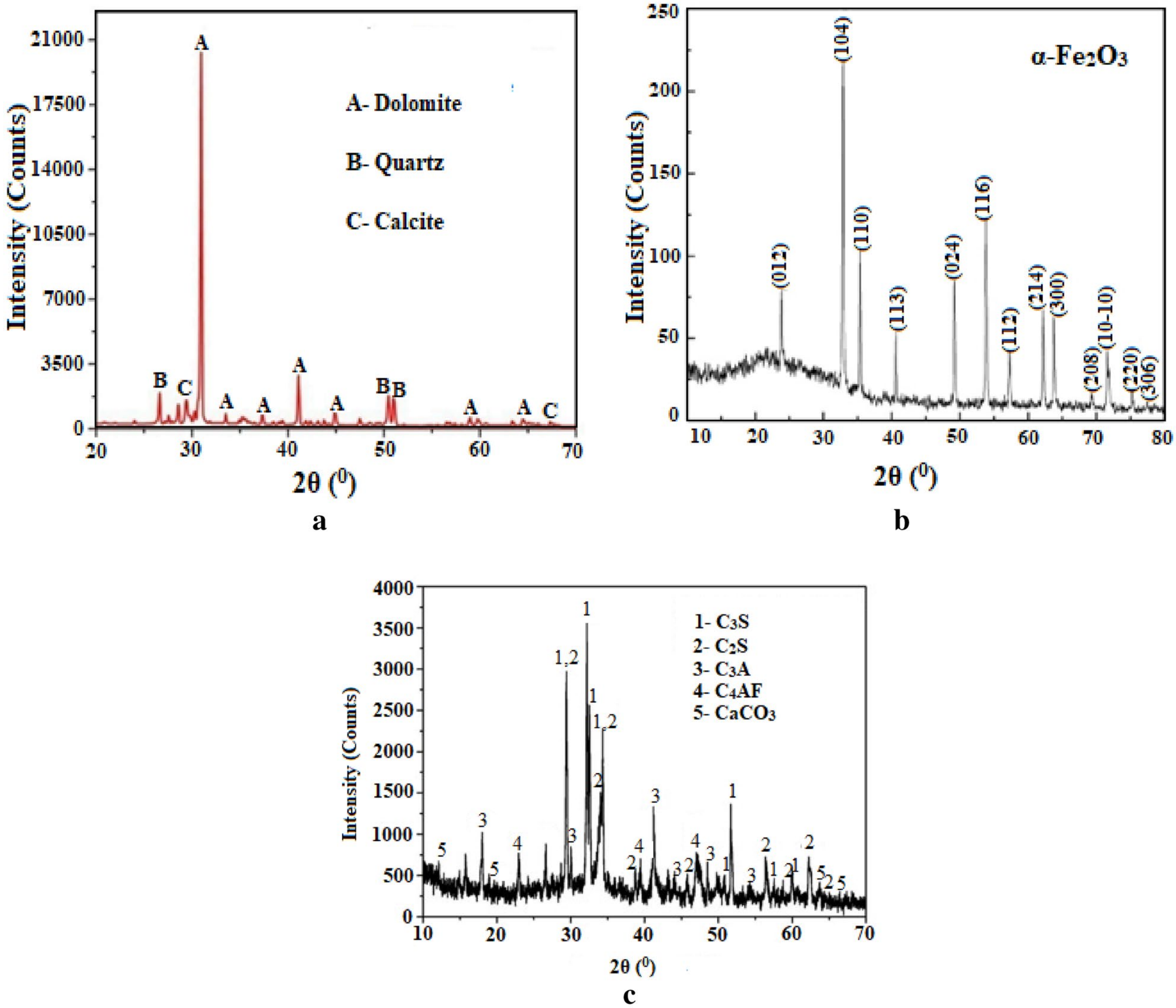


Fig. 1 **a** XRD pattern of MP, **b** XRD pattern of NF. **c** XRD pattern of OPC

The mold was removed to measure the flow spread size and the average of five measurements were noted.

2.3.2 Compressive Strength Test

The compressive strength test was performed on7th, 14th, and 28th day by following ASTM C109-16 (ASTM, 2015b) procedures. The compressive strength investigation of the cubic specimens size of 50 mm × 50 mm × 50 mm was performed utilizing (AIM 302E-FA-1EM). The compressive strength was determined using average strengths of the three examined specimens.

2.3.3 Split Tensile Strength Test

ASTM C496-16(ASTM, 2017) procedure was adopted for examining the split tensile strength. The cylindrical specimen having 100 mm diameter and 200 mm height of 7th, 14th and 28th day cured aged samples were investigated. A tensile strength testing machine (50-C9000/C) was used to apply longitudinal stress to cylindrical samples at a 1 mm/min loading rate.

2.3.4 Characterization of Materials

The Scanning Electron Microscopy (SEM) test was performed using JEOL-JSM-IT 200 with EDS. The morphology, size, and material characteristics of MP, NF, and cement in powder form were investigated. In a high vacuum chamber, test specimens mounted on platinum-coated carbon adhesive tape underwent characterization analysis using a voltage of 15 kV.

2.3.5 XRD Investigation

The X-ray diffraction (XRD) technique was performed to characterize the samples of MP, NF, and cement. The powder form of samples was placed inside the sample holder and mounted on a monochromatic (Ge-220) Triple axis (Xe) detector. The sample analysis was performed at room temperature with 45 kV with 40 mA while using the exploration range of 2θ.

2.3.6 Water Absorption Test

Water absorption test was done in accordance with ASTMC1403-15 to investigate the durability of cement mortars (ASTM, 2015c). The specimens were prepared using the same processes for mixing, casting, and compaction for compressive and split tensile tests. The samples were de-molded and then placed vertically within a watertight container. The samples from the 28th day were taken out of the container and heated to 100 °C for 24 h. The heated samples were allowed to cool at normal temperature. The initial weight of samples was weighed (0.01 g), and then the specimens were immersed in another container having approximately 3 mm height of water. To assess the amount of water absorbed, the sample was weighed after 15 min, 30 min, 1, 2, 3, 4, 6, 24, 48, 72, 96, 120, and 144 h. The water absorption values of the experimental samples at various intervals were calculated using the equation represented below and the average of 3 samples measurement was recorded:

$$\mathrm{A}_t = \frac{(W_T - W_0)}{A} \times 100$$

where A_t, W_T, W_0, and A, respectively, stand for intake uptake, sample weight at a particular time (T), Initial sample weight, and cross-sectional area.

2.3.7 Rapid Chloride Iron Penetration Test (RCPT)

The cylindrical samples of control and blended mortar samples of size 100 mm in diameter and 20 mm in height were tested for their resistance to chloride-ion penetration after 28th day of curing according to ASTM C1202-12 (ASTM C, 1202–12 2012). The mortar samples were subjected to RCPT with the use of a PVC container and a 60 V impressed voltage. The positive and negative terminals of the cells were filled with 0.3 N NaOH and 3% concentrated NaCl solution, respectively. Every 30 min for up to 6 h, the current flow was monitored and noted. Using time and current consumption, the chloride ion permeability was determined in terms of coulombs.

2.3.8 Chloride Diffusion Coefficient

The entire migrated of chloride ion particles were measured in mortar specimens after 28 days of curing. Until a steady state was reached at 120 h, the samples were regularly aquilots to determine the chloride concentration of the sample. Nernst–Einstein equation (Song et al., 2009) has been used to find the coefficient of chloride diffusion:

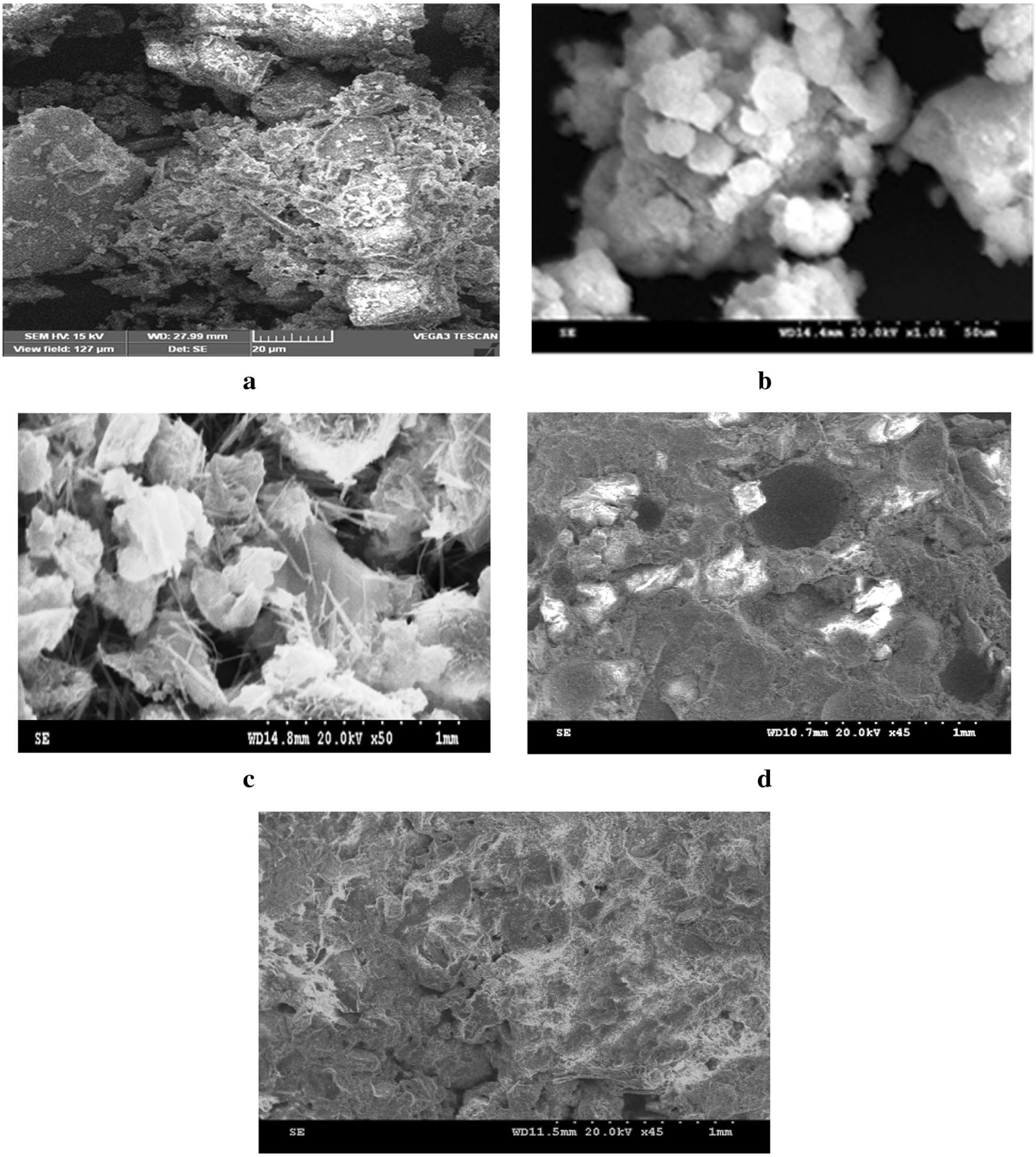
$$D = \frac{JRTL}{ZFC_0E}$$

where D is the Diffusion coefficient of chloride (m^2/s), J is the chloride ions flux (mol/m^2 s),Z is the chloride ion valency ($Z=1$), R is constant of gas (8.314 J/K mol), L is the Specimen thickness (cm), T is absolute temperature (300 K), F is the Faradays co-efficient (9.648×104 J/V mol), C_0 is the chloride ion concentration (initial) (mol/l), and E is the applied potential voltage (60 V).

3 Results

3.1 Characterization of Materials

The morphology and characterization of the samples were examined using Scanning Electron Microscope (SEM). Fig. 2a, b shows the SEM image of MP and NF. The average MP particles are smaller than 20 µm and sum larger particles are with fine clay particles are in agglomerated. Majorly, the structure of the particle was mainly composed of lamellar particles without pores. The NF particles are crusted together with spherical shaped crystals with particle size of less than 5 nm size. The XRD patterns of MP is shown in Fig. 1a which indicates that the major component was calcium oxide with minor component of silicon dioxide and oxides of magnesia with the indication of carbonate nature, quarts were also identified at very low concentration. Fig. 1b shows the XRD humps of NF, it shows the strong atomic ratio of the iron oxide nanoparticles without the presence of other elements. At 2θ = 35.66°, the peak with the highest intensity was noted. The JCPDS-86–0550 readings, which can be connected with the (012), (104), (110), (113), (024), (116), (112), (214), (300), (208), (010), (220), and (306) planes of hematite (α-Fe_2O_3), verified the diffraction peaks' appearance at various angles (2θ).This demonstrated that there are no additional contaminants or other types of iron oxide phases present in the final particles, which are pure α -Fe_2O_3. Fig. 1c exhibits the XRD humps of OPC at 2θ. It was found that the highest peaks of Alite, Belite with calcite and also found with medium peak of ferrite with aluminate and calcium carbonate. Fig. 2c exhibits the SEM image of cement paste, it shows plate and needle shaped CH hydrates with C–H–S. The SEM picture of a cement and marble powder paste, which is denser and has fewer pores, is shown in Fig. 2d at this stage exhibits; calcium silicate hydrate (C–S–H) in the



e

Fig. 2 **a** Morphology of MP. **b** Morphology of Nano Fe_2O_3. **c** SEM image of OPC. **d** SEM image of OPC + MP. **e** SEM image of OPC + MP + NF

form of composed amorphous particles and massive layers of calcium hydroxide crystals are appeared. Ettringite spines are visible in pores, the voids are fully compacted and paste was fully hydrated. SEM image of OPC with MP and NF is illustrated in Fig. 2e. The microstructure of the paste appeared denser than OPC with MP paste; meanwhile, hydrated products can be found more compact with higher density and more structured C–S–H gel. The significant change in this stage was the formation of thick ettringite crystals in pores. They were entangled and crossed over one another to form a complicated network, which lower the porous nature.

3.2 Effect of MP and NF Particles on Mortar's Fresh and Hardened Properties

The results of flow table test, which was used to assess the flow ability of fresh mortar mix are shown in Fig. 3. The effects of the MP and NF on fresh characteristics were varied. The addition of 5%and 10% of MP increased the flow spread by 2.07% and 3.18% compared to control mix. This might be because of the lower specific gravity of MP than cement. Similar trend was reported in previous studies, where cement was replaced by MP (Yamanel et al., 2019). When the dosage was reached at 15%, the flow trend exhibited a decreasing nature. In 20% dosaged sample 6.84% decrement of flow spared compare to 10% MP dosaged sample was observed, but the workability was in the acceptable range. The addition of high amount of marble powder in cement mortar causes increase of friction and decrease of workability corroborating with the previous reports (Ashish et al., 2016; Toubal Seghir et al., 2019). In the second phase, the mortar mix was dosaged with NF-1and NF-2, which showed a flow spread increment of 3.89–4.95% compared to control mix. 1.5% (NF-3) dosaged sample exhibited a decreasing trend with 5.05% less flow spread compared to NF-2. However, all the dosages are within the acceptable limit and NF showed more workability in all test concentrations rather than MP dosage.

The MP and NF blended cement mortar specimens strength were examined by compressive and tensile strength tests, and the results are shown in Table 2. Both MP and NF samples showed a general strength improvement tendency up to their ideal limits of 10% and 1%, respectively; after this percentage of replacement, it showed a strength decreasing trend. Initially the strength increased up to 3.80% and 6.47% with MP 5% and 10%, respectively. Beyond 10%, a sudden drop in strength was observed. It might be because of a drop in cementing materials C_3S and C_2S in mortar mix, which are mainly responsible for cement strength. NF dosaged samples showed the strength increment of 5.71% and 12.40% compared to the control mix at 0.5%, 1% dosage levels; meanwhile, exceeding 1% dosaged strength decrement trend was observed. However, MP dosaged samples recorded lesser strength compared to NF dosaged samples.

Table 2 Flowability and ultimate strength

Sample ID	W/B	Flow Spread (mm)	Compressive strength-28th day (MPa)	Split Tensile strength-28th day (MPa)
C-1	0.5	283	33.38	2.46
MP-5	0.5	289	34.65	2.51
MP-10	0.5	292	35.54	2.59
MP-15	0.5	286	32.74	2.36
MP-20	0.5	273	29.72	2.04
NF-1	0.5	292	35.28	3.19
NF-2	0.5	297	37.54	3.27
NF-3	0.5	282	29.42	1.94
NF-4	0.5	271	24.68	1.52

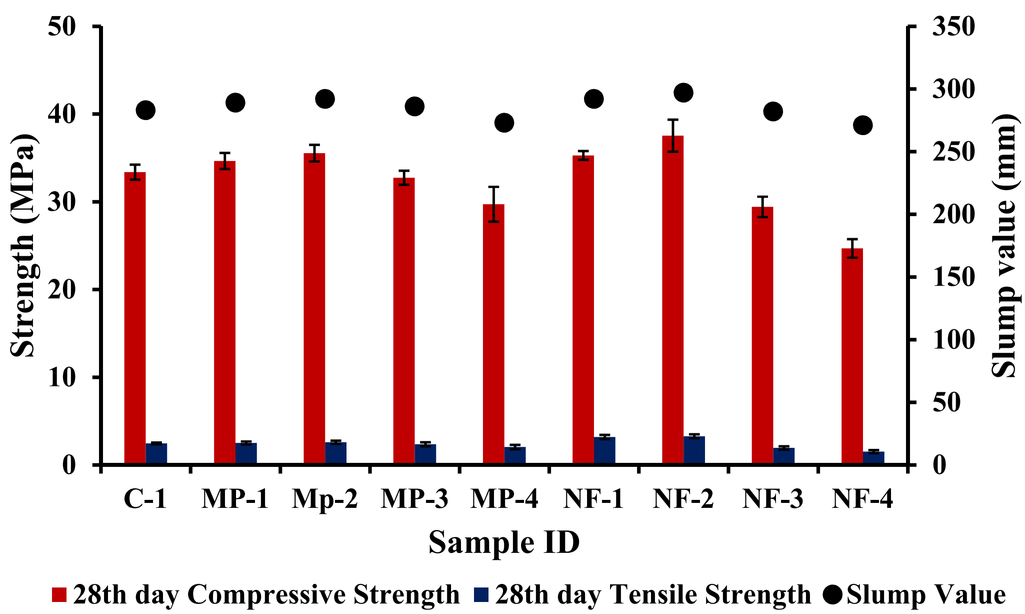


Fig. 3 Ultimate strength and slump

The results of split tensile strength are shown in Table 2; a general strength improvement trend was for both MP and NF dosage samples. A maximum split tensile strength of 2.59 MPa, 3.27 MPa was recorded for 10% MP and 1% NF, respectively. Here also we observed that the addition of NF provides additional strength compared to MP dosaged samples. However, further than the optimum amount NF showed sudden strength decrement than MP dosaged samples. It was noted that the split tensile strength is greatly impacted by the presence of nanoparticles. Huge quantities of nanoparticles added resulted in poor tensile strength and low interfacial or interface properties. From the observation, the optimum dosage of MP and NF particles for further study was fixed as 10% and 1%, respectively.

3.3 Effect of Optimized Combined Dosage of MP and NF on Fresh as Well as Strength Development (MPNF)

From the standardized study results, this same ideal limit of MP and NF was observed to be 10% and 1%, respectively. At the intervals of 7, 14, and 28, MPNF samples' strength growth was tested. The 7th, 14th, and 28th days were exhibits increases in compressive strength of 71.42%, 34.20%, and 23.60%, respectively. The findings demonstrated that the combined addition of MP and NF nanoparticles, rather than the individual dose is more effectively enhances the final strength. However, early age strength growth was improved significantly with MPNF particles.

The split tensile strength development of MPNF samples, which showed higher strength instead of control as well as independent dosages of MP and NF. On the 7th, 14th, and 28th days of the interval, the overall strength increased by 79.03%, 36.70%, and 41.46%, respectively. Therefore, the split-tensile strength is significantly improved by the addition of MPNF particles.

3.4 Water Absorption Test

The capillary water input is mostly responsible for mortar's durability. The control, individual optimal dosages of MP, NF samples are absorbed more water rather than MPNF blended samples. This noticed that two phases of water absorption and the values are shown in Fig. 4. The optimum dosage of (10% MP and 1% NF) specimens sucked up less water compared with control specimens. In contrast, the MPNF blended samples absorbed far less water than the other specimens. In initial stage, water up take of Control, optimum dosage of MP and NF samples are high, but in secondary stage (after 24 h) the MP and NF dosage samples absorbed less water compared to initial the stage. The results proved that addition of MP and NF makes the micro structure denser and reduced the pore holes especially, the combination of MP and NF, which enhanced the durability rather than other samples.

3.5 Rapid Chloride Iron Penetration Test (RCPT)

The Rapid Chloride Penetration Test (RCPT) has been used to evaluate the durability of cement mortar against chloride ingress. Table 4 illustrates the RCPT results at the end of the 28th day for mortar with optimum replacement of MP, NF and combined dosage MPNF. MP in cement mortar had lesser Coulomb values compared to

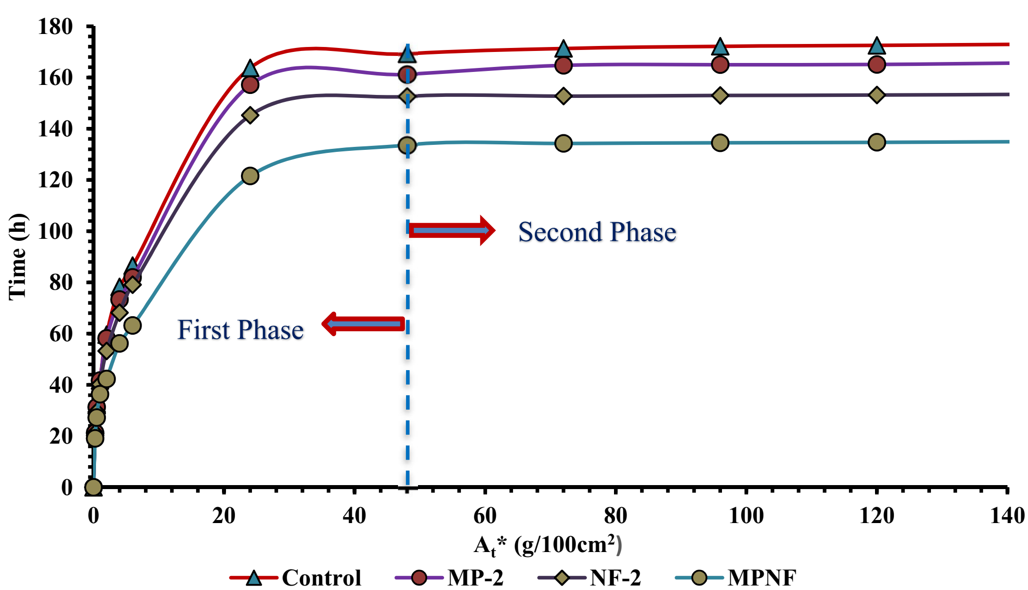


Fig. 4 Rate of water absorption

control mix. However, NF in cement mortar significantly decreased the chloride penetration compared to control. For example, in control mortar, the charge passed Coulomb value is 2577, while in the optimum NF dosage mortar and MPNF, the charge passed Coulomb values are333 and 312.3 Coulomb, which was around 87.07% and 87.88% lower than control mortar.

3.6 Chloride Diffusion Coefficient (Condition-Steady State)

The chloride diffusion coefficient of mortar specimens at steady sate condition after 28th day curing was shown in Table 3. The result showed that the samples of MP-2 and NF-2 mortar mix significantly reduced the chloride diffusion. Such as, MP-2 samples scored 1.48×10^{-5} m/s and NF-2 scored 2.79×10^{-7} m/s this was lesser than 2.07×10^{-5} m/s, 3.52×10^{-5} m/s compared to control mix. Meanwhile, the combination of MPNF reduced the chloride diffusion but the rate of reduction is not high compare to NF-2 dosage. It may be because of the high reactivity of MP particles that easily absorb the moisture and convert to $Ca(OH)_2$, which in turn react with atmospheric CO_2, to form solid $CaCO_3$that fills the pores and voids of cement paste. On the other hand, NF produces a structure that is more densely bound and modifies the pore structure while increasing the viscosity of the cementitious matrix.

4 Discussion

4.1 Preliminary Studies

The inclusion of MP and NF has a gradual impact on the mechanical properties of cement-based composites, according to the screening studies. The compressive strength of MP and NF was gradually increased by 10% and 1%, respectively. For 1% of NF dose samples, the split-tensile strength improved dramatically. The specimens with NF showed greater strength than those with MP indicating that the inclusion of 1% nanoparticles particles results in greater strength than that of the inclusion of 10% MP particles. This might be attributable to NF particles' superior packing capacity when compared to MP particles. Moreover, the result showed similar to previous studies, cement mortar with nano particles have denser and more compacted and also the microstructure appears to be the most homogeneous with the least porosity for the optimum dosage of nanoparticles included (Ding Siang Ng, 2020). Nano reinforcing materials are created when nano ferric oxide reacts with calcium hydroxide to density the concrete microstructure (Heikal et al., 2021). The addition of NF allows for more C–S–H nucleation sites, which speeds up the hydrated products growth in the pore spaces and voids of concrete materials thereby directly enhancing the properties of cement mortar.

Table 3 Chloride diffusion coefficient

Sample Id	Charge passed (coulomb)	Free chloride (ppm)	Flux of chloride ions (J)	Steady state diffusion coefficient (D) m^3/s
Control	2577	1276	8.47×10^{-5}	3.55×10^{-5}
MP-2	2143	886	5.88×10^{-5}	1.48×10^{-5}
NF-2	333	319	2.11×10^{-5}	2.79×10^{-7}
MPNF	312	283	1.88×10^{-5}	8.89×10^{-8}

Moreover, the introduction of nanoparticles beyond optimum concentration results in agglomeration leading to a decrease the strength, which was found to be greater than 1% for both compressive and tensile. The high surface energy of nanoparticles causes agglomeration and irregular dispersion of nanoparticles in the matrix resulting in the generation of weak zones in the mortars (Kong et al., 2012).As a result of this agglomeration, some of the oxygen groups on the surface of NF nanoparticles gets entangled, rendering them unreachable to the matrix's wet ability and preventing them from making a contribution to hydration and interaction with C–S–H bonds (Keller et al., 2010). The C–S–H structure in cement mortar with iron oxide (IO) desperation was noted to be significantly greater than that of agglomerated IO (Sikora et al. 2016). Meanwhile, the high amount of nano particles absorbs more water thereby increasing the water demand as well as affecting the workability. Previous studies have also reported that the formation of weak zone due to agglomeration as the level of NF (> 3%) is increase (Ding Siang Ng, 2020). However, in this investigation, it is reported that strength loss if NF amount is more than 1% of cement weight; which could be due to the inadequate level of C–H in the hydrated products to react with rise in ferrous content.

The maximum flow recorded for MP-1 is 293 mm, but NF-2 exhibits 290 mm, although the difference was quite modest and within the permissible range. Fig. 3 illustrates growing flowability with an increase in particle dose up toward the optimal limit. Meanwhile, MP dosage samples always had greater flowability than NF dosage samples. The NF particles high surface area directly influences workability, which in turn raises the amount of water in an indirect way. The strength decline is clearly apparent despite the 20% MP and 2% NF dose specimen workability to be within acceptable limits. However, the workability was unchanged. The mechanical qualities were seen to improve in samples with 10% MP and 1% NF.

4.2 Strength Development of Optimized Samples

The compressive and split tensile strength of MP-2 samples are high after 28th day curing period compare to other MP dosage samples. The compressive strength increases, because marble powder particles act like filler material, which leads to form denser mortar mix by filling the void spaces. The filler effect enhances cement matrix as well as transition zone characteristics (Ashish et al., 2016).Two major minerals of marble powders are calcium carbonate and calcium magnesium carbonate, in addition, small quantities of alumina and silica are also present. Calcium silicate hydroxide ($Ca(OH)_2$) and silica may react, to increase the compressive strength. The outcomes are consistent with the earlier investigations, where (Toubal Seghir et al., 2019) Marble powder's CaCO reacts with cement's tricalcium aluminate(C_3A), and produces calcium carbo-aluminate thereby enhancing the strength and the binding property of concrete. NF-2 samples having 1% of optimum dosage scored higher mechanical property compared to MP-2 as well as control samples. The strength growth was gradually increased at the curing period of 7th, 14th, and 28th days, which clearly demonstrates strong integration among both C–S–H crystals and NF particles has been accompanied by a rise in the nanoparticle dosage while increasing the speed of hydration. Similar effects were also observed in previous research (Singh et al., 2017). However, after 7th days, the strength growth slowed, maybe because there is not enough space for the growth of $Ca(OH)_2$ crystals (Omar et al., 2012).

4.3 Strength Development of MPNF Specimens

The combined effect of (MPNF) dosage cement mortar strength was investigated using an optimum limit of MP and NF. The strength development of MPNF samples, which was similar to the NF additive samples, which it demonstrates early age strength development. The 7th day compressive strength test findings of MPNF specimens recorded 41.76% higher than MP-2 samples and 16.67% higher than NF-2. The combined effect of MP (MP-2) and NF (NF-2) promoted strength development by filling the inner pores, rate of hydration in cement and enhanced the C–S–H production especially in comparison with other mixes. However, the split tensile strength of MPNF samples were significantly increased, which might be because of the combination of MP and NF increased C_3S, which modified C–S–H and enhanced the potential crystallization.

In addition to the figures and tables showing the difference between the control and experimental results; statistical significance was also checked especially for the compressive and tensile results comparing with control. Among the different statistically significant test methods, one way ANOVA was performed in SPSS 17.0 software using Tukey's post hoc analysis with *P* value less than the alpha (α) value that is 0.05 or 5%, the goodness of fit was represented as scatter plots, Fig. 5. The results indicate that upon the inclusion of additives (NF and MPNF), both the compressive and split tensile strength showed a significant ($p < 0.05$) increase in strength compared to the control.

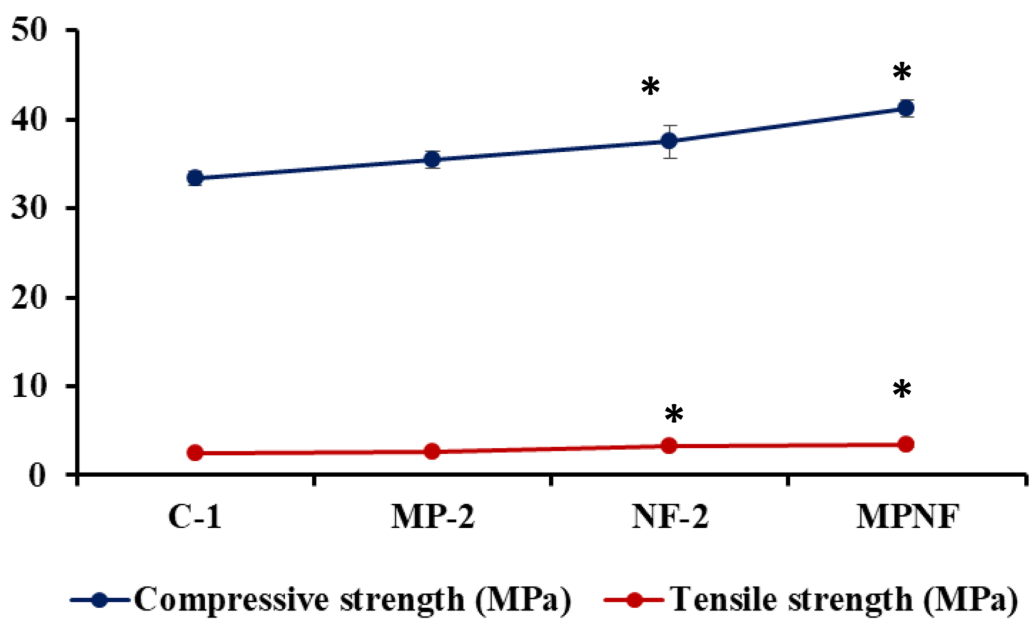


Fig. 5 ANOVA results; the significance of control and experimental results with different replacements (* Indicates significance at $p < 0.05$ when compared between NF-2 and MPNF vs other mix design groups)

4.4 Durability of Optimum Dosage Samples

Durability is the important factor of cementations composites. The combination of MP and NF reduced the water penetration rather than individual dosage samples 22.8%, 13.72% compared to optimum dosage of MP and NF, respectively. The reason for this improvement is because the MP fills the pore holes of inner matrix and NF refines the pores and makes cement matrix can get densely packed by reducing the amount of free water (Amer et al., 2015; Li et al., 2006).

RCPT results are shown in Table 4, which indicates that MP-2 and NF-2 dosage samples significantly enhanced the chloride resistance property compared to optimum dosage samples as well as control. The result showed a decreasing trend with optimum combination of MP and NF (Control > MP > NF > MPNF). This might be because of the low amount of alumina in the marble powder, which promotes the formation of tricalcium aluminate that favors fixation of the chloride ions. Meanwhile, NF particles make strong bridge between crystals of C–S–H with MP particles, which can accelerate the hydration to make a contribution toward the chemical resistance and also changes the interior structure of C–S–H gels, so it leads to stabilized calcium irons. These ions are more stable and reduce the chloride diffusion, this is in agreement with previous report were the Nano iron oxide particles accelerated the hydration leading to chloride resistance (Heikal et al., 2021; Khoshakhlagh et al. 2012)

5 Conclusion

The efficiency of cement mortar upon the introduction of marble powder (MP) as well as Nano ferrous (NF) at varying concentrations levels individually and their combined effect was evaluated using flow table test, compressive, split tensile and durability tests. The results demonstrated that the individual effects of MP and Nano Fe_2O_3addition on mechanical and fresh qualities were distinct. In general, MP dosage mixes showed increase in water content because of their high fineness. Meanwhile, particles increased the workability up to optimum limit, and also increased the rate of hydration, refined the pores and enhanced C–S–H gel formation showing better strength improvement rather than MP dosage. However, comparison to control and individual effects of MP and NF, the cement mortar's strength and durability were improved by the combination of MP (10%) and NA (1%). The study findings support the use of MP and NF in concrete technology by showing that partial cement replacement with marble powder and nano-Fe_2O_3 is effective in enhancing properties of cementitious composites.

Authors' information

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Author contributions

JPA: writing—review/editing, formal analysis, and data collection. AGA: Conceptualizations, formal analysis, investigation, design, Experimentation, writing original/final draft, writing—review/editing visualization supervision and project administration. TG: experimentation, writing—review/editing, formal analysis, and data collection SM: writing—review/editing, formal analysis, and data collection. All authors read and approved the final manuscript.

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Data availability

All the data associated with this study are available from the corresponding author upon request.

Declarations

Ethics approval and consent to participate

All authors of the manuscript confirm ethical approval and consent to participate following the Journal's policies.

Consent for publication

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Competing interests

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