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RESEARCH

Use of Nano Silica Sol for Performance Improvement of Marine Cement-Based Mortar

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ABSTRACT

Nano silica sol is an aqueous solution containing colloidal nano-silica. From limited research in the literature, it has been reported to offer better effects on the cement-based materials compared with nano-silica powder, which is by far the most widely studied nano-material. In this research, the possible use of nano silica sol to improve the performance of marine cement-based mortar is investigated. The optimal type of nano silica sol among acidic, neutral, and alkaline pH is identified, followed by systematic experimentation of the flexural strength, compressive strength, chloride ion permeability, as well as Fourier-transform infrared (FTIR) spectrum and X-ray diffusion (XRD) pattern. It was found that alkaline nano silica sol outperformed acidic and neutral counterparts. Generally, the addition of nano silica sol increased the flexural and compressive strengths of marine mortar. Addition of 3% nano silica sol by volume of cement yielded remarkable effects, while further addition of nano silica sol yielded diminishing returns. The FTIR spectra and XRD patterns suggested the nano silica sol induced nucleation effect at relatively early age and pozzolanic reaction effect at relatively late age. These effects improved the performance of marine cement-based mortar.

KEYWORDS

Colloidal nano silica, Chloride ion permeability, Marine cement-based mortar, Nano silica sol, Nucleation effect.

INTRODUCTION

In tandem with the increase in construction volume of marine and offshore structures, there has been growing demand of marine concrete and mortar that is better able to withstand the aggressive marine environment than conventional concrete and mortar. Compared to inland environment, marine exposure is mainly characterised by the frequent of wetting and drying as well as abundance of chloride ion, which are deleterious to the depassivation and corrosion of reinforcement bars, and hence impairing the durability of marine and offshore structures [**1**]. According to recent survey, a large proportion of seaport projects constructed with conventional concrete suffered from substantial dilapidation after 8 to 10 years of service [**2**]. This invoked significant depreciation of engineering asset and repair cost. Therefore, the development of marine concrete and mortar with good performance is of crucial importance.

Vast studies have shown that the incorporation of nano-silica powder in cement systems could promote cement hydration, improve the microstructure of cement-based matrix, and enhance the mechanical properties and durability of cement-based composites [**3-9**]. However, it has been acknowledged that the nano-silica powder is very difficult to achieve uniform dispersion throughout the cement-based mixture [**10**]. To tackle this challenge, the potential use of nano silica sol is hereby explored.

Nano silica sol is an aqueous solution containing colloidal nano-silica. It can exist in stable state and is miscible with water, thereby practically resolving the problem of evenly dispersing nano-silica powder. From limited research in the literature on the application of nano silica sol [**11-13**], it has been reported to offer better effects on the cement-based materials compared with nano-silica powder. Overall, there is a lack of systemic studies on the roles of nano silica sol in the mechanical properties and durability of cement-based mortar and concrete. In-depth research along this direction is thus needed. Particularly, the possible properties enhancement for marine construction is investigated in the present works.

In general, dependent on the applications, the pH of nano silica sol can be adjusted. Hereunder, the effects of acidic, neutral and alkaline nano silica sol on marine cement-based mortar are first studied, so as to identify the optimal type of nano silica sol. The performance improvements including the flexural and compressive strengths as well as the chloride ion permeability of marine mortar are quantified. The paste phase of mortar is characterised via Fourier-transform infrared (FTIR) and X-ray diffusion (XRD) analyses to reveal the roles of nano silica sol in cement hydration and chemical reactions.

EXPERIMENTAL CAMPAIGN

Raw materials

In the experimental campaign, both marine mortar and paste samples were prepared in accordance with the test requirements. The cement used was a marine class cement complying with Chinese Standard GB/T 31289-2014. It contained no more than 0.06% chloride ion and no more than 4.5% sulphur trioxide, and the detailed chemical composition is listed in **Table 1**. Its density was 3020 kg/m^3.

The nano silica sol contained silica content of 30%, with the size of silica particles ranging from 10 nm to 20 nm and possessing a density of 1020 kg/m^3. According to the manufacturer, the nano silica sol was prepared with three pH ranges: acidic nano silica sol with pH from 2 to 4; neutral nano silica sol with pH from 6 to 8; and alkaline nano silica sol with pH from 9 to 11. The actual pH values of the acidic, neutral, and alkaline nano silica sol were measured to be 4, 7, and 10, respectively.

Table. 1. Chemical composition of marine class cement.

Chemical composition	Mass percentage
Silicon dioxide	27.5
Calcium oxide	49.8
Aluminium oxide	8.59
Magnesium oxide	4.67
Sulphur trioxide	2.68
Ferric oxide	2.16
Potassium oxide	0.83
Sodium oxide	0.65

The fine aggregate for mortar was standard sand conforming to International Standard ISO 679-2009. Its moisture content, water absorption, fineness modulus, and particle density were measured to be 0.31%, 0.99%, 2.37, and 2420 kg/m^3, respectively.

Superplasticizer was added to attain proper workability of the mortar. The superplasticizer was polycarboxylate-based aqueous solution conforming to Chinese Standard GB 50119-2013. The mixing water was from ordinary potable water source.

Sample preparation

Mortar samples and paste samples were prepared to suit the test requirements. Regarding mortar sample, the mix proportions are summarized in **Table 2**. The nano silica sol content was varied from 0 to 5% by volume of cement. The mixing sequence and protocol were according to Chinese Standard GB/T 17671-2021. The water/cement ratio was fixed at 0.50 by mass and the aggregate-to-cement ratio by mass was set as 3:1. The superplasticizer dosage was adjustable to achieve a consistent workability of 200±10 mm slump flow. The fresh mortar was cast into prismatic steel moulds of dimensions 160 × 40 × 40 mm (for strength measurement) and 100 × 100 × 50 mm (for chloride ion diffusion coefficient measurement), stored indoor and demoulded after 24 hours, then cured in standard curing room until the specified testing age.

Table. 2. Mix proportions of mortar samples.

Mix ID	Type of nano silica sol	Nano silica sol content (%)	Super-plasticizer dosage (%)	Slump flow (mm)
CTM	Nil	0	0	200
AM1	Acidic	1	0.20	205
AM2		2	0.50	200
AM3		3	0.80	195
AM4		4	1.50	190
AM5		5	2.00	190
NM1	Neutral	1	0.10	205
NM2		2	0.20	195
NM3		3	0.40	190
NM4		4	1.00	190
NM5		5	2.00	190
LM1	Alkaline	1	0.05	205
LM2		2	0.10	195
LM3		3	0.30	190
LM4		4	0.80	195
LM5		5	1.80	190

Regarding paste sample, the mix proportions are tabulated in **Table 3**. The paste sample represented the paste portion of the mortar sample, with the nano silica sol content varied among 0 and 3% by volume of cement, and the water/cement ratio fixed at 0.50 by mass. The fresh paste was placed into cubic steel moulds and demoulded after 24 hours, then cured in standard curing room until the age of testing. The handing of nano-materials in the laboratory has conformed to the health and safety requirements in relevant International Standard ISO/TR 12885-2018.

Table. 3. Mix proportions of paste samples.

Mix ID	Type of nano silica sol	Nano silica sol content (%)	Super-plasticizer dosage (%)
CTP	Nil	0	0
AP3	Acidic	3	0.80
NP3	Neutral	3	0.40
LP3	Alkaline	3	0.30

TEST METHODS

Flexural and compressive strengths

The measurement of flexural strength and compressive strength of the mortar mixes was conducted as per stipulations in Chinese Standard GB/T 17671-2021. The flexural and compressive strengths at age of 3-day, 7-day and 28-day were determined. At each age of testing, three 160 × 40 × 40 mm mortar prisms were loaded under three-point bending configuration with a span of 100 mm until failure, and the averaged result was taken as the flexural strength. The two broken halves from each prism were subjected to compression test, and the averaged result from six broken halves was taken as the compressive strength.

Chloride ion permeability

The chloride ion permeability of mortar mixes was determined in accordance with Chinese Standard JC/T 1086-2008 in terms of the chloride ion diffusion coefficient. Mortar prisms of size 100 × 100 × 50 mm were cast and cured in standard curing room until attaining 28-day age, immersed in a tank containing saturated sodium chloride solution with suction pressure applied to the air space above the liquid level over a duration of 18 hours, then the chloride ion diffusion coefficient (in m^2/s) was tested using bespoke testing instrument (model: NELD-CCM540). A smaller coefficient value indicates a lower chloride ion permeability and hence better durability, and vice versa.

Fourier-transform infrared analysis

To carry out the analysis, paste samples were taken from the curing room at the age of 1-day and 7-day, crushed into fragments and soaked in pure ethanol for 7 days to cease hydration. The fragments were then dried inside an oven maintained at 40°C to constant weight, and ground to passing 75-micron aperture sieve. The sieved powder was analyzed using FTIR spectrometer (model: FTIR-650) with a spectral wavelength ranging from 4000 cm^{-1} to 400 cm^{-1} and a resolution of 4.0 cm^{-1}.

X-ray diffraction analysis

To conduct the test, paste samples were taken from the curing room at the age of 1-day, 7-day, and 28-day, crushed into fragments and soaked in pure ethanol (the procedures of preparing the fragments are the same as those for FTIR analysis, including the ensuing drying, grinding and sieving processes). The sieved powder was analyzed through X-ray diffraction with the starting angle to ending angle set as 2° to 80°, the tube voltage rated at 40 kV and the tube current rated at 40 mA.

RESULTS AND DISCUSSIONS

Flexural strength

The flexural strengths of marine cement-based mortar incorporating three types of nano silica sol (acidic, neutral, alkaline) at age of 3 days, 7 days, and 28 days are plotted in **Fig. 1**. For the control mortar mix without incorporating nano silica sol (mix CTM), the strength development with time was reflected by the flexural strength results of 4.8 MPa at 3 days, 5.4 MPa at 7 days, and 6.3 MPa at 28 days. At all ages, the use of acidic nano silica sol caused reduction of flexural strength compared to the control mortar mix. In contrast, the neutral nano silica sol mildly reduced the 3-day flexural strength, slightly increased (at 4% nano silica sol content) or decreased (at other contents than 4%) the 7-day flexural strength, and slightly increased the 28-day flexural strength. Regardless of age, the use of alkaline nano silica sol increased the flexural strength, where the ranges of flexural strengths at 3-day, 7-day, and 28-day were respectively 4.8 to 5.2 MPa, 5.5 to 6.2 MPa, and 6.6 to 6.9 MPa.

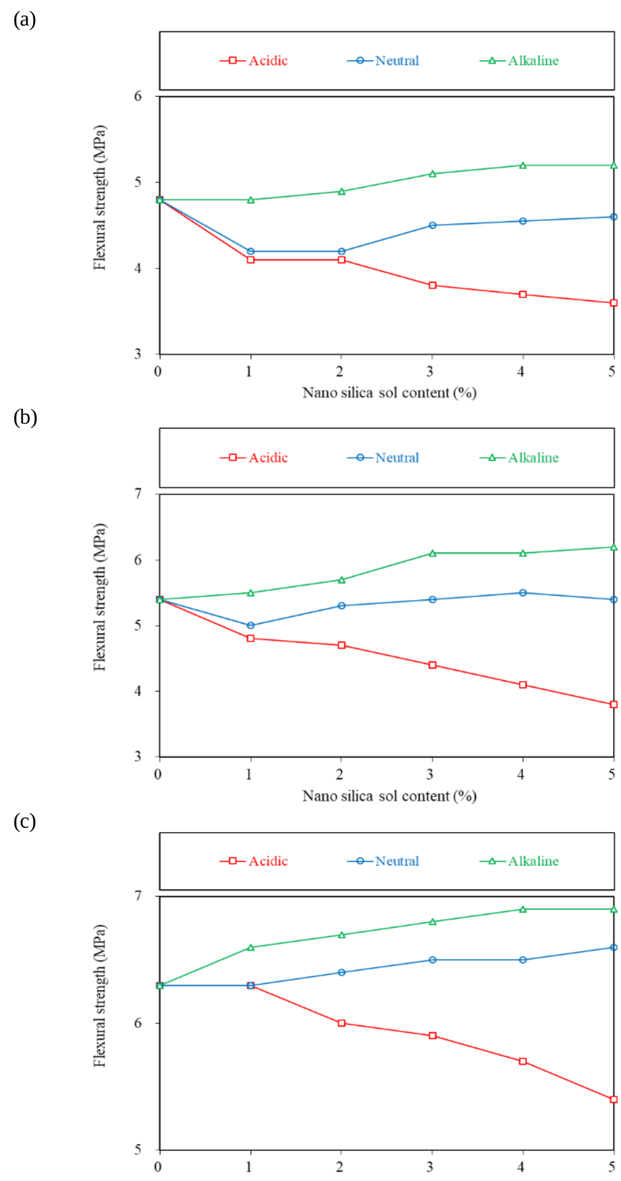


Fig. 1. Flexural strength of nano silica sol marine mortar: (a) 3-day, (b) 7-day and (c) 28-day.

The peak percentage increase in flexural strength is identified as 14.8%, which was brought about by adding 5%

alkaline nano silica sol at 7-day age. In-depth inspection of the results revealed diminishing return beyond 3% alkaline nano silica sol content, where the percentage increase in flexural strength was 13.0%. At 28-day age, the percentages increase in flexural strength by adding 3% and 5% alkaline nano silica sol were respectively 7.9% and 9.5%. Thus, the alkaline nano silica sol was more beneficial to the early (7-day) flexural strength. This could be due to the nucleation effect of nano silica sol, where the nano-sized silica particles act as nuclei to promote hydration of cement at early age.

Compressive strength

The compressive strengths of nano silica sol marine cement-based mortar at 3-day, 7-day, and 28-day age are plotted in **Fig. 2**. For the control mortar (mix CTM), the strength development with time was reflected by the compressive strength results of 22.4 MPa at 3 days, 33.2 MPa at 7 days, and 44.2 MPa at 28 days. At all ages, the acidic nano silica sol could decrease (at 1% and 2% contents) or increase (at higher than 3% contents) the compressive strength. The use of neutral nano silica sol at small contents (1% and 2%) marginally reduced the early-age compressive strength, while usage at larger contents (3% and above) increased the compressive strength at any age. Regardless of age, the use of alkaline nano silica sol increased the compressive strength.

The experimental results of neutral and alkaline nano silica sol marine mortar are inspected quantitatively. For the neutral pH group of mixes, the ranges of compressive strengths at 3-day, 7-day, and 28-day were respectively 20.0 to 26.4 MPa, 30.9 to 37.3 MPa, and 43.4 to 49.7 MPa, and the peak percentage increase in compressive strength was 17.9% by adding 5% neutral nano silica sol at 3-day age. Whereas for the alkaline pH group of mixes, the ranges of compressive strengths at 3-day, 7-day, and 28-day were respectively 22.5 to 28.1 MPa, 33.5 to 39.3 MPa, and 47.9 to 51.6 MPa, and the peak percentage increase in compressive strength was 25.4% by adding 5% alkaline nano silica sol at 3-day age. It is noted that the percentages increase became smaller at 7-day and 28-day age. Thus, the beneficial effects of neutral and alkaline were more apparent on the very early (3-day) compressive strength. This is in line with previous findings [**13**] and would be due to the nucleation effect of nano silica sol that accelerates cement hydration as mentioned in the preceding section.

Among all the mixes, the effectiveness of nano silica sol in enhancing compressive strength of marine mortar followed the descending order: alkaline nano silica sol > neutral nano silica sol > acidic nano silica sol. The reason would be due to the less compatibility of acidic and neutral nano silica sol in the inherently alkaline environment of cement-based mortar, possibly leading to agglomeration of the nano-sized silica particles and hence diminution of the beneficial effects [**14**]. Last but not least, the use of alkaline nano silica sol beyond 3% content exhibited diminishing return in improving the compressive strength. For instance, at 3-day age, the percentage increase in compressive strength by adding 3% alkaline nano silica sol was 23.7% (versus 25.4% by adding 5% alkaline nano silica sol); at 28-day age, the percentages increase in compressive strength by adding 3% and 5% alkaline nano silica sol were respectively 15.8% and 16.7%.

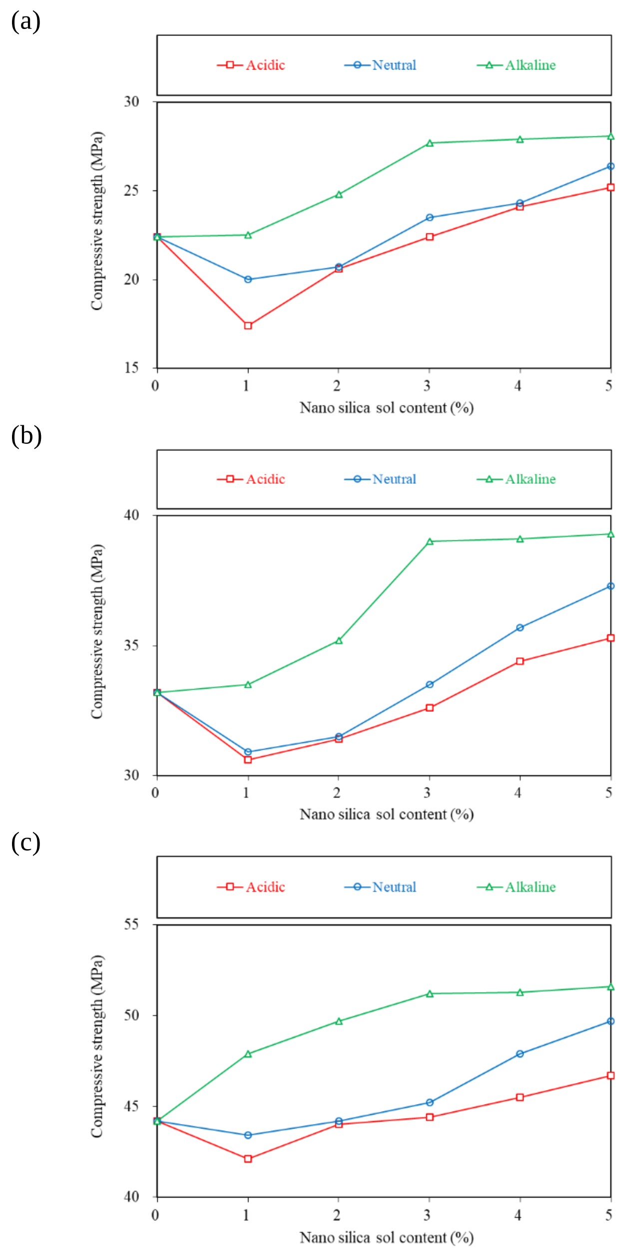


Fig. 2. Compressive strength of nano silica sol marine mortar: (a) 3-day, (b) 7-day and (c) 28-day.

Chloride ion diffusion coefficient

From the above experimental results, it is evidenced that the alkaline nano silica sol could best improve the mechanical properties of marine cement-based mortar. Therefore, alkaline nano silica sol is chosen for further study of its effect on the chloride ion permeability. **Fig. 3** depicts the experimentally obtained chloride ion diffusion coefficient versus the alkaline nano silica sol content. It can be seen that the control mortar mix without nano silica sol (mix CTM) had the largest coefficient value (1.601×10^{-12} m^2/s) and hence possessing the highest chloride ion permeability among the series of mortar mixes. The chloride ion diffusion coefficient decreased with increasing alkaline nano silica sol content until reaching the minimum value (0.818×10^{-12} m^2/s) at 3% alkaline nano silica sol content, then turned to increase with the nano silica sol content while the coefficient value remained less than that of mix CTM.

Overall speaking, alkaline nano silica sol is able to reduce chloride ion permeability and improve durability. The alkaline silica sol content of 3% could be regarded as optimal, where the chloride ion diffusion coefficient was 48.9% lower than that of the control mortar. The possible mechanism could be the nano-sized silica particles reduced the micro-pores throughout the cement paste matrix and interfacial transition zone between the cement paste and fine aggregates, thereby densifying the microstructure of mortar and lowering the permeability [**15**].

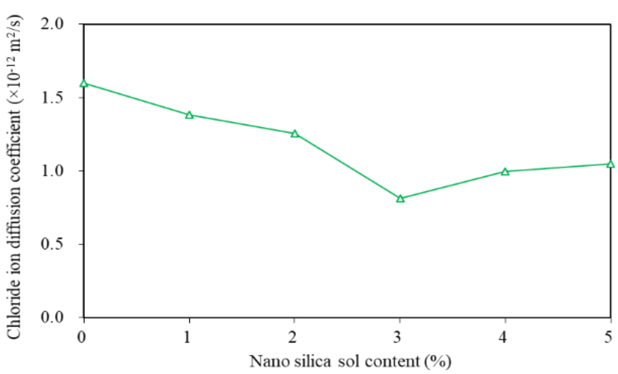


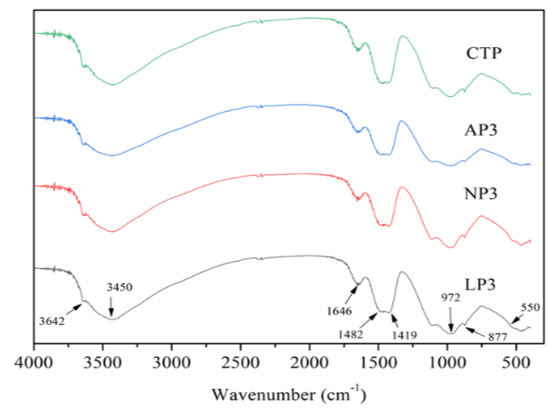
Fig. 3. Chloride ion diffusion coefficient of nano silica sol marine mortar.

Fourier-transform infrared spectra

Paste samples containing 3% nano silica sol (the optimal content in connection with the minimum chloride ion diffusion coefficient) were adopted for undergoing FTIR and XRD analyses. To examine the effects of different types of nano silica sol on the hydration and chemical reactions, the control paste mix without incorporating nano silica sol (mix CTP) and paste mixes at different pH levels (mixes AP3, NP3, LP3) were analysed. The FTIR spectra at age of 1-day and 7-day are displayed in **Fig. 4**. From the spectra, the wavenumber of 3642 cm^{-1} corresponds to the O-H vibration of the hydration product $Ca(OH)_2$; the wavenumbers of 3450 cm^{-1} and 1646 cm^{-1} correspond respectively to the stretching mode and bending mode of H-O-H vibration from the combined water of the hydrated gel product; the wavenumber around 1419 cm^{-1} and 877 cm^{-1} correspond respectively to the symmetrical stretching vibration and twisting vibration of C-O possibly resulted from the carbonation of hardened cement paste during curing and/or fragment drying and grinding; the wavenumber around 550 cm^{-1} corresponds to stretching vibration of Al-O, indicating the presence of Aft ettringite; the wavenumber around 972 cm^{-1} corresponds to the asymmetric stretching vibration of Si-O, resembling the characteristic peak of C-S-H gel [**16-17**].

As observed from **Fig. 4**, in comparison with the control paste mix, the absorption peak of C-S-H gel notably had higher intensity in the presence of nano silica sol. This could be due to the nucleation effect of nano silica sol in promoting hydration of cement and formation of C-S-H gel at early age, and is in agreement with reported findings [**12**].

(a)



(b)

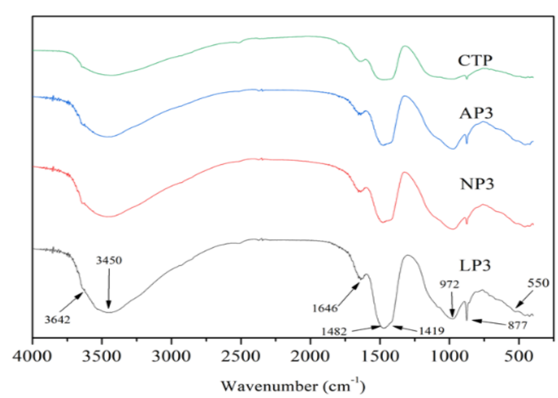


Fig. 4. FTIR spectra of nano silica sol marine mortar: (a) 1-day and (b) 7-day.

X-ray diffraction patterns

The XRD patterns of paste mixes at age of 1-day, 7-day, and 28-day are displayed in **Fig. 5**. At 1-day age, compared with the control paste mix, the characteristic peaks for $Ca(OH)_2$ of mixes AP3 and NP3 were heightened moderately, while the characteristic peaks for $Ca(OH)_2$ of mix LP3 was heightened significantly, indicating the nucleation effect of nano silica sol to promote cement hydration at early age. At 7-day and 28-day age, the intensity of characteristic peak for $Ca(OH)_2$ of mix LP3 decreased abruptly to become less than other mixes. On the other hand, the characteristic peak for C-S-H was heightened substantially. This could be explained by the pozzolanic reaction effect of nano silica sol to chemically react with $Ca(OH)_2$ to form hydrated calcium silicate gel. Moreover, the characteristic peaks for dicalcium silicate (C_2S) and tricalcium silicate (C_3S) of mix LP3 had lower intensities than those of mixes AP3 and NP3. This could be due to the promotion of conversion of C_3S by alkaline nano silica sol.

It can be seen from **Fig. 5** that the XRD patterns at 7 days and 28 days of age exhibited substantial similarities. The reason would be the nano silica sol exerted major influence on cement hydration during the first 7 days of age. This is in line with the research findings that silica sol promotes cement hydration primarily at early age, while it has little influence on cement hydration at late stage [**18-19**]. Finally, it is evidenced from the above analysis that alkaline nano silica sol could better promote cement hydration and chemical reactions than acidic and neutral nano silica sol.

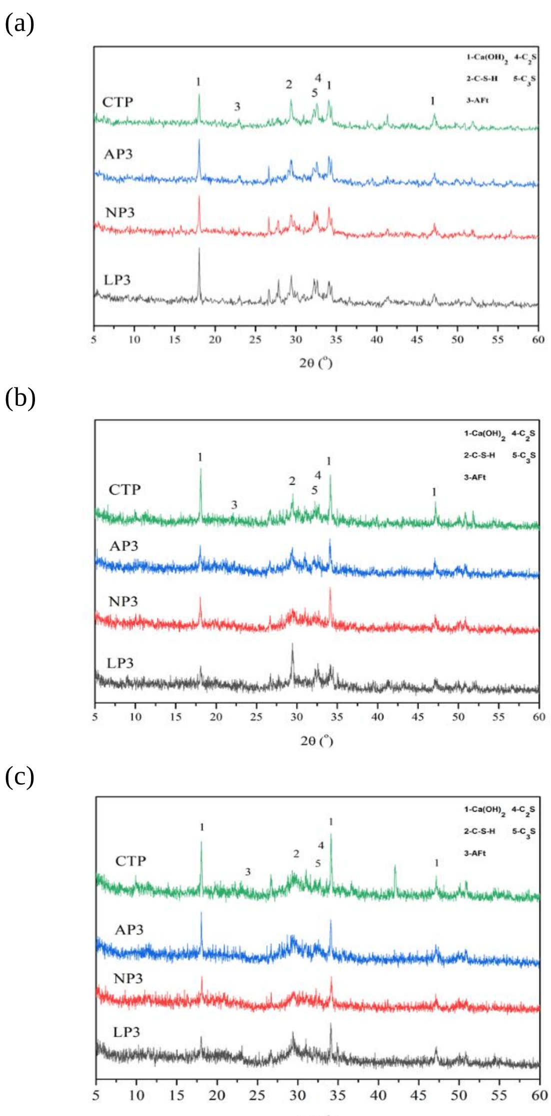


Fig. 5. XRD patterns of nano silica sol marine mortar: (a) 1-day, (b) 7-day and (c) 28-day.

CONCLUSION

From the experimental works reported hereinabove, the conclusions can be drawn:

(1) The effectiveness of nano silica sol in increasing the strength of marine cement-based mortar follows the descending order: alkaline nano silica sol > neutral nano silica sol > acidic nano silica sol. The peak percentage increase of flexural strength was 14.8% (at 7-day and 5% alkaline nano silica sol) and peak percentage increase of compressive strength was 25.4% (at 3-day and 5% alkaline nano silica sol). On the other hand, the percentages increase corresponding to 3% nano silica sol were respectively 13.0% and 23.7%. This evidenced the diminishing returns of nano silica sol beyond 3% content.

(2) Upon increasing the content of alkaline nano silica sol, the chloride ion permeability of marine mortar first decreased and then increased. At 3% nano silica sol, the chloride ion diffusion coefficient was the minimum, and was 48.9% lower than the control mortar mix. Further increasing the nano silica sol content resulted in slight and gradual increase in the chloride ion diffusion coefficient, indicating that the optimal content of alkaline nano silica sol should be 3%.

(3) Through FTIR (Fourier-transform infrared) and XRD (X-ray diffraction) analyses, it was revealed that the nano silica sol consumed the calcium hydroxide generated from cement hydration and produced additional C-S-H gel. The FTIR spectra and XRD patterns indicated the nucleation effect of nano silica sol at relatively early age and pozzolanic reaction effect at relatively late age.

Overall, the use of nano silica sol could improve the performance of marine cement-based mortar. Further research on blended usage of nano silica sol with various binder materials in mortar mixes is recommended.

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CONFLICTS OF INTEREST

There are no conflicts to declare.

SUPPORTING INFORMATION

Supporting informations are available online at journal website.

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Authors Biography

Dr. P.L. Ng obtained his Bachelor of Civil Engineering and Doctor of Philosophy degrees at the University of Hong Kong. He is chartered civil and materials engineer and is licensed in Hong Kong, United Kingdom, and Europe. He has more than 20 years of experience in research on concrete materials, and is a committee member of the Hong Kong Institution of Engineers Materials Division. He has published more than 180 scientific papers and delivered more than 75 technical speeches. His works and contributions have been recognised by a number of prestigious awards.

GRAPHICAL ABSTRACT

Compressive strength and chloride ion diffusion coefficient of nano silica sol marine mortar at 28-day age (*top left and right*); Fourier-transform infrared (FTIR) spectrum and X-ray diffraction (XRD) pattern of nano silica sol marine mortar at 7-day age (*bottom left and right*).