



# Combined effect of metakaolin and sea water on performance and microstructures of concrete



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## HIGHLIGHTS

- The compressive strength of concrete was improved by metakaolin and seawater.
- There is a relationship between average pore diameter and compressive strength.
- Chloride was immobilized by formation of Friedel's salt to increase chloride resistance.

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## ABSTRACT

The effect of 0–6 wt% MK and mixing with seawater on the properties, hydration and microstructure of concrete was studied. The compressive strength at 28 days increased by 33% when addition of 5 wt% MK and by 22% when mixed with seawater. The combination of both increased compressive strength by 52%. The pore structure was refined under both conditions. There is a relationship between average pore diameter and compressive strength. MK promoted the formation of Friedel's salt in concrete mixed with seawater. MK and mixing with seawater improved chloride resistance. This study shows that MK and seawater improved the performance of concrete.

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## 1. Introduction

Concrete structures consumed considerable amount of Portland cement, which resulted in substantial amount of carbon dioxide during the heating calcite above 1450 °C to produce one of the main raw materials, calcium oxides [1]. To reduce the carbon footprint and save energy without reducing performance, supplementary cementitious materials (SCMs) are introduced into concrete to substitute part of cement since 1950s. SCMs are mostly pozzolanic materials including industry wastes such as fly ash and slag [2–5].

Pozzolanic materials, especially silicate fume, slag and fly ash, have been widely used in construction. Metakaolin (MK) has the similar effect to improve strength and durability of concrete as silicate fume [6,7]. For the last several decades, the influence of MK on the properties of concrete were studied by a range of researchers, and the results confirmed that MK is a concrete admixture

with high activity and high performance improvement [8–17]. MK is produced by calcining kaolin at 650–800 °C [18,19]. The main components of MK, which are amorphous  $Al_2O_3$  and  $SiO_2$ , have high pozzolanic activity. Besides the filling effect, MK reacts with calcium hydroxide (CH), which is one of the hydration products of Portland cement, to form calcium silicate hydrate (C–S–H) gels, the main binding phase of concrete [20,21]. CH was considered to be the main factor leading to the weakness of interfacial transition zone (ITZ) which in turn influences the strength, pore structure and durability of concrete [2]. The addition of pozzolanic materials consumes CH and improves the performance of concrete. As a porous material, the pore size distribution and structure of concrete affect the properties and durability of concrete. The addition of MK refines the pore structure of concrete and so as to improve the strength and durability of concrete [8,22].

Replacing fresh water with seawater during the construction of concrete structure in coastal areas will reduce the cost of transportation and increase the work efficiency. However, according to some previous researches, the use of seawater leads to a shorter

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service life of buildings, where the rapid corrosion of rebar became the main failure factor. In MK modified concrete, chloride ions can be immobilized by formation of Friedel's salt to improve the corrosion resistance of concrete, which makes the use of MK in marine concrete beneficial.

The cost of MK is much higher than that of other SCMs. Currently, most researches focused on the concrete containing 10–30 wt% MK. In this study, the properties and microstructure of concrete with 0–6 wt% MK addition and artificial seawater substituting fresh water were characterized by a range of analytical techniques to assess the feasibility of low MK addition and seawater mixing in concrete.

## 2. Materials and experimental

### 2.1. Materials

Type P.I. 52.5 Portland cement was used for all concrete and paste mixes. MK was obtained by calcining kaolin at 750 °C. The chemical composition of cement and MK was characterized by X-ray fluorescence (XRF) (Table 1). Local natural fine aggregate in a maximum size of 4.75 mm, and coarse aggregate in a maximum size of 26.5 mm were used. Artificial seawater was prepared according to the standard ASTM D1141-98 [23] (Table 2).

### 2.2. Specimen preparation

Concrete mixes were designed at water/binder ratio of 0.45. The percentages of MK that replaced cement in this study were 0%, 2%, 3%, 4%, 5% and 6% by mass. Mix design of concretes and pastes are shown in Tables 3 and 4. The density of concrete was 2400 kg m<sup>-3</sup>. After 24 h curing in room temperature and relative humidity of 100%, the concrete and paste specimens were demolded and cured at 20 °C.

### 2.3. Test methods

#### 2.3.1. Compressive strength

Concrete cubes in size of 100 × 100 × 100 mm and paste cubes in size of 40 × 40 × 40 mm were prepared for compressive strength test after cured for 3, 7 and 28 days at 20 °C. Three specimens were tested for each batch.

#### 2.3.2. Pore size distribution

Specimens from the central part of concrete cubes were used for mercury intrusion porosimetry (MIP) test after cured for 3, 7 and 28 days. Specimens were immersed in ethanol to stop hydration after crushed and dried at 105 °C for 24 h before test.

#### 2.3.3. X-ray diffraction (XRD)

XRD was performed on freshly ground paste specimens by Bruker D8 Advance under the conditions of Cu K $\alpha$  = 1.5406 Å, step size of 0.019°, measuring time 141.804 s/step, start position 5° and end position 70°. The acquired data was analyzed by PANalytical X'pert Highscore Plus with PDF2004 database.

#### 2.3.4. Scanning electron microscopy (SEM)

SEM was performed on concrete specimens by FEI Quanta 450FEG under the conditions of spotsize 5 and accelerating voltage 20 kV. Hydrated specimens were cut into thick slices and then hydration stopped by immersion in isopropanol before being embedded in resin, then polished and coated with carbon.

#### 2.3.5. Fourier transformation infrared spectroscopy (FTIR)

FTIR was employed to analyze the chemical bonding of hydration products by Thermo Nicolet Nexus FTIR under Attenuated Total Reflectance (ATR) mode from 4000 cm<sup>-1</sup> to 400 cm<sup>-1</sup>.

Specimens for FTIR test are the same as those for XRD test. Based on the peak of calcium hydroxide at 3640 cm<sup>-1</sup>, the content of calcium hydroxide of each specimen was calculated by deconvoluting overlapped peaks [24]. A typical FTIR spectrum of specimen is shown in Fig. 1.

### 2.3.6. Chloride penetration

Concrete specimens were prepared in cylinders of 100 mm in diameter and 150 mm in height and cured at 20 °C. After cured for 28, 56 and 70 days, the specimens were cut into pieces of 100 mm in diameter and 50 mm in height. The chloride penetration properties were tested according to the standard ASTM C1202.

## 3. Results and discussion

### 3.1. Compressive strength

The compressive strength of concretes and pastes are shown in Figs. 2 and 3 respectively.

For concrete mixed with fresh water, the compressive strength increased with the time of hydration. For the mixes at the same age, the compressive strength increased with content of MK and reached the maximum at 5 wt% of MK. The highest compressive strength was obtained by C5 at 28 days, which increased by 33% comparing to C1 at the same age. The compressive strength of the concretes mixed with seawater (C7–C12) had a similar trend to that of concretes mixed with fresh water. The compressive strength increased by 24% for C11 comparing to C7. Some research showed that addition of 5 wt% MK did not influence the compressive strength of concrete at 28 days. However, some other research indicated that the compressive strength increased by 25% with the addition of 5 wt% MK [25–28]. The difference may be due to the various composition and microstructures of MK from different sources.

The compressive strength of concrete mixed with seawater was higher than that of the concrete mixed with fresh water under the same content of MK and same age. The combination of MK and seawater effectively improved the mechanical performance of concrete. According to the results and discussion later in this study, the reason is that seawater accelerated the hydration of cement to increase the content of CH available for pozzolanic reaction with MK, which resulted in more C–S–H formed in the concrete.

The development of relative strength of concretes comparing to the control specimen C1 is shown in Fig. 4, together with those from other researchers [9,29–33]. The trend of change of relative strength from this study is similar to those from other researchers.

For specimens mixed with fresh water, the relative strength reached the maximum by 28 days and decreased gradually but was still higher than the control. In this study, addition of 5 wt% MK increased the compressive strength by 33% at 28 days. For the addition of 2–30 wt% MK, the compressive strength increased by 5–35% at 28 days according to Fig. 4. Although the content of MK is relatively low in this study, the compressive strength increase is considerable.

For specimens mixed with artificial seawater, the relative strength reached the maximum by 7 days and decreased gradually but still much higher than the control and fresh water specimens at the same age. Addition of 5 wt% MK and mixing with seawater greatly improved the compressive strength of concrete by 56%, 52% and 46% at 7, 28 and 56 days respectively.

Fig. 3 shows the compressive strength of paste, which had the similar trend to that of concrete.

### 3.2. Pore structure

The effects of seawater and MK on the pore structure of concrete are studied by MIP, and the results are shown in Figs. 5–7

**Table 1**

The chemical composition of cement and MK characterized by XRF (%).

Oxides	Na <sub>2</sub> O	MgO	Al <sub>2</sub> O <sub>3</sub>	SiO <sub>2</sub>	P <sub>2</sub> O <sub>5</sub>	SO <sub>3</sub>	K <sub>2</sub> O	CaO	Fe <sub>2</sub> O <sub>3</sub>	LOI <sup>a</sup>
Cement	0.09	1.60	4.15	19.05	0.09	3.32	0.77	64.43	3.29	2.43
MK	0.39	0.07	38.63	57.37	0.61	0.15	0.49	0.03	0.77	1.04

<sup>a</sup> LOI: loss on ignition.

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