

## Increasing mortar strength with the use of activated kaolin by-products from paper industry

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### ABSTRACT

Making use of industrial by-product or waste clay to partially replace cement in concrete has greatly contributed to sustainable development of environment. This study investigated the optimal activation condition for producing high reactivity metakaolin (MK) by using kaolin by-products (KB) from paper industry. Initially, the material properties of KB were analyzed in this study and the results indicated its great potential to be treated by calcination to be a very effective pozzolan. Afterwards, MK samples produced from different activation treatments of KB, including different calcining temperature, calcining duration, initial temperature rise time and grinding particle size, were applied in mortar to determine their pozzolanic effect. Results indicated that the optimal activation condition for KB to high reactivity MK conversion is 2-h calcining duration at 750 °C and with 7 μm grinding particle size. Finally, compressive and flexural strengths of mortar samples produced by replacing cement with 0–25% MK content were tested and the results showed that 15% was the optimal cement replacement level of MK and the mortar so produced exhibited 20% improvement in compressive strength compared to control mortar.

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### 1. Introduction

Concrete, a man-made product with about six billion tons consumption every year, is the most extensive used construction material in the world and only second to water as the most heavily consumed substance. This is largely due to the abundance of raw materials for cement manufacture, relatively low cost and the versatility and adaptability of concrete with various shapes. However, damage of environment caused by the extraction of raw material for cement production and global warming problem due to the CO<sub>2</sub> emission during cement manufacture have resulted in heavy pressures to reduce cement consumption. In order to lower the cement consumption, using environmental-friendly supplementary materials to partially replace cement should be of great concern [1]. These environmental-friendly supplementary materials could be naturally available materials, industrial wastes or industrial by-products and those require relatively less energy to manufacture [2].

Literatures have shown that the utilization of calcined clay in the form of metakaolin (MK) as a pozzolanic partial cement replacement material for mortar or concrete has received wide interest in recent years. A majority of this interest has come to the removal of CH, the product of the hydration of cement, which is related to the poor durability of mortar or concrete. The CH re-

moval can improve the ability of resistance against sulphate attack and alkali reaction (ASR) and can also grant enhanced strength which is derived from the additional cementitious materials generated by reaction of CH and MK [3–12]. This excellent pozzolanic property of MK is characterized by its layered silico-aluminate, in which the interlayer water loses after calcination and becomes a high pozzolanic combination of Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub>. This combination in an active form will react with the CH. High reactivity MK can be obtained by carefully heating kaolin clay to temperatures at 600–800 °C [13–22]. Currently, it is usually manufactured by firing the high-grade purified kaolin; however, its application in regular concrete is rather limited due to relatively high production costs.

Kaolin by-products (KB) are the coarser part of kaolin particles sieved out from paper mill with size above 0.02 mm. It accounts for nearly 30% of kaolin in paper industry and usually served as a rejected material, which aggravates environmental stress and actually leads to a waste of resource. As the KB has a high content of Al<sub>2</sub>O<sub>3</sub> and SiO<sub>2</sub> and fineness similar to that of Ordinary Portland Cement (OPC), it is the authors' belief that KB with suitable calcining and grinding treatment may be used as a kind of pozzolanic additive to increase the strength of mortar or concrete and to solve the disposal problem of this industrial waste for the sake of sustainable environment at the same time. This paper aims to investigate the optimal activation treatment process for KB to obtain a high reactivity pozzolanic additive and the optimal cement replacement level of this activated material so produced to improve the strength of mortar.

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## 2. Experimental program

### 2.1. Materials used

KB used in this study was supplied from a local paper mill. In the paper manufacturing, kaolin clays were classified according to its fineness using decanter centrifuge. The finer part was used for making paper after chemical bleaching treatment, pressure filtration and spray drying process, while the coarser part with an average size larger than 0.02 mm was collected and pressed to be KB lump.

The OPC (CEM I 42.5R) used in this study was tested to conform to Chinese standard code GB175-1999 [23]. Its initial setting time was tested to be 140 min after water addition and its compressive strength was tested to be 19.4, 32.6, 44.5 and 52.2 MPa at the age of 1, 3, 7 and 28 days according to EN 196 [24].

The fine aggregate used in this study was graded river sand passing through 1.18 mm sieve. Its fineness modulus and specific gravity were measured to be 2.84 and 2.52, respectively.

### 2.2. Material characterization of KB

Chemical composition of KB was analyzed using X-ray fluorescence (XRF) technique and the analysis results (Table 1) showed that KB contains 38.33%  $\text{Al}_2\text{O}_3$ , which is higher than the common range (15–30%), and 46.98%  $\text{SiO}_2$ , which is lower than the common range (55–80%). It is noteworthy that the ratio of  $\text{Al}_2\text{O}_3/\text{SiO}_2$  for KB is 0.82 and very close to the theoretical value (0.85) for the unitary cell of kaolinite ( $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ), which shows the predominance of the clay mineral in KB [25].

Mineralogical analysis of KB was examined by X-ray diffraction (XRD) with a 36 kV, 20 mA Cu radiation source and the diffraction results are shown in Fig. 1. It can be observed that the six fingers-shaped peaks located at  $2\theta$  between  $35^\circ$  and  $40^\circ$ , representing the characteristic peak of kaolinite ( $\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2 \cdot 2\text{H}_2\text{O}$ ). Two sharp and symmetrical crystallization peaks corresponding to  $12.5^\circ$  and  $25.1^\circ$  ( $2\theta$ ) represent good crystallinity of kaolinite which shows the predominance of kaolinite mineral again and hence indicating a high pozzolanic potential for KB [19]. A peak at  $27.36^\circ$  ( $2\theta$ ) represents the quartz crystal phase of  $\alpha\text{-SiO}_2$ . According to calculation of semi-quantitative mineralogical estimation, the quartz content of  $\alpha\text{-SiO}_2$  in KB is nearly 13%, which may somehow impair the pozzolanic reactivity potential of KB.

The physical parameters of KB, including the specific surface area, specific pore volume and pore diameter, were measured by a surface area and porosimetry system as  $8.5922 \text{ m}^2 \text{ g}^{-1}$ ,  $0.027295 \text{ mL g}^{-1}$  and  $15.7125 \text{ nm}$ , respectively. The results are shown in Table 1. Besides, the morphological analysis of KB was also conducted using scanning electron microscopy (SEM) technique and the photo is shown in Fig. 2. It can be observed that KB has a particle size of roughly  $2 \mu\text{m}$  and closely packed layer structure with high crystallinity.

**Table 1**  
Chemical and physical properties of KB.

Chemical composition (% w/w)								
$\text{SiO}_2$	$\text{Al}_2\text{O}_3$	CaO	MgO	$\text{Fe}_2\text{O}_3$	$\text{K}_2\text{O}$	$\text{Na}_2\text{O}$	$\text{SO}_3$	L.O.I
46.98	38.33	0.01	0.42	1.08	0.82	0.01	0.28	9.2
Physical characteristics								
Specific surface area ( $\text{m}^2 \text{ g}^{-1}$ )							8.5922	
Specific pore volume ( $\text{mL g}^{-1}$ )							0.027295	
Pore diameter (nm)							15.7125	

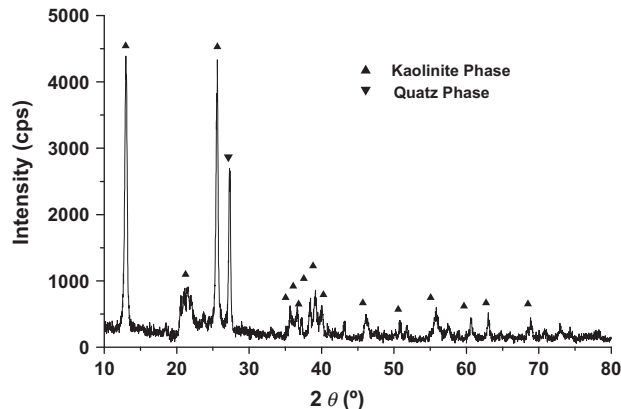


Fig. 1. XRD analysis result of KB.

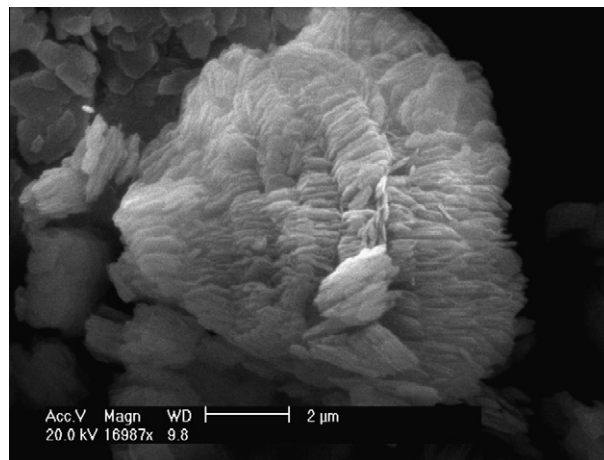


Fig. 2. SEM image of KB.

Thermal decomposition property of KB was measured by differential thermal and thermogravimetry analysis (DTA–TG) and the results are shown in Fig. 3. It can be observed that the dehydration stage for KB lies between  $450^\circ\text{C}$  and  $700^\circ\text{C}$ . The kaolinite–metakaolin endotherm is observed at  $553^\circ\text{C}$  with a weight loss of 10.2%. This provides the estimated kaolinite content of 80.5%. The sharp exothermic peak at  $1004^\circ\text{C}$  as shown in DTA curve demonstrates the change of the phase, i.e., formation of spinel phase. Thus, a rough estimation can be drawn that the calcination temperature for completing the KB–MK transformation is within  $500\text{--}900^\circ\text{C}$ .

Based on the foregoing material properties investigations, it can be seen that KB has a high content of kaolinite and shows the predominant characters of the kaolinite mineral. Thus, there would be a great potential to treat KB by thermal activation to be an effective mineral additive.

### 2.3. MK sample preparation

To find out the optimal activation treatment process for completing KB to high reactivity MK conversion, KB was treated under different calcination conditions in a laboratory electrical furnace (380 V, 12 KW, 50 Hz and value error within  $\pm 0.3^\circ\text{C}$ ) with the abundant supply of air and grinding conditions in a laboratory ball mill (QM-2SP20-CL) with revolution speed and rotation velocity of 180 and 270 rpm, respectively. The treatment conditions tested include:

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