



# Characterization of metakaolin and study on early age mechanical strength of hybrid cementitious composites



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## HIGHLIGHTS

- MK can be produced in the laboratory at 800 °C for 1 h.
- MK, CNS and epoxy resin at 10%, 1% and 1% respectively reduces slump value and increase the density as the ages increase.
- The flexural strength increases at all ages with incorporation of natural and synthetic fibres.
- The relationship between the compressive strength and flexural strength is significant.

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## ABSTRACT

In this study, the potency of using laboratory produced metakaolin as partial replacement of cement in the evolution of a hybrid cementitious composites (HCC) was investigated. Some chemical composition and phases of metakaolin were appraised with the aid of X-ray fluorescence (XRF) and X-ray diffraction. Particle size analyzer was used to study the physical properties of metakaolin produced in the laboratory from purified Kaolin. Early age mechanical strength and shrinkage properties of structural grade HCC developed by replacing fly ash in the original ECC M45 design with metakaolin (MK), colloidal nanosilica (CNS) and epoxy were examined. The designed mixtures consist of five mixtures with the control and base mix exposed to water and seawater. The control mix consists of only the fine aggregate and cement while the base mix consists of cement, fine aggregate, 10% MK and CNS with epoxy resin at 1% each by weight of the binder. Synthetic barchip fibres and some natural fibres namely, oil palm fruit bunch and coconut fibres were incorporated. The water cement ratio was designed as 0.30 for all mix ratios. The result reveals that the inclusion of MK, CNS and epoxy have a significant effect on the fresh properties of the HCC and at early age of 7, 28 and 90 days. The mechanical strengths were better improved than the standardized M45 ECC at these early ages.

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## 1. Introduction

The impressive headway in the development of highly improved and performance cementitious composite materials cannot be an oversight in these late years. This includes numerous of high strength concrete usually produced with low water binder ratios such as pultruded continuous fibre reinforced concrete which was pioneered by Mobasher et al. [1]. High performance fibre reinforced cementitious composite which has a unique improved strength as easily as high ductility due to a strain hardening response [2], Eco friendly concrete that contain an increase by-products and mineral admixtures which make it to be more

environmentally friendlier than the conventional concrete. Green high performance cementitious (GHPC) material is developed in a decade ago as established by Zhongwei [3]. The most important features of GHPC is the use of several admixtures to replace cement content partially [4]. Similarly, this category of concrete material consists of a matrix that does not contain coarse aggregates hence regarded as concrete materials. The most acceptable and widely use mineral admixtures are silica fume (SF), fly ash (FA) and ground granulated blast furnace slag (SL) which are byproducts of industrial process according to Zhu [5]. Nonetheless, other cementitious materials like metakaolin is a natural pozzolanic material derived from calcinated clay would have bigger potential for enhancing engineering properties in concrete.

Engineered Cementitious Composites, ECCs as usually called, is a class of high performance fibre reinforced composites and also a green high performance cementitious material due to the nature of

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cement replacement. It is typified by its unique excellent strain capacity under uniaxial tension, with a controlled fibre volume fraction usually 2% or less [6]. ECC is similar to a ductile metal, hence has the capacity to strain harden after first crack with a demonstrated strain capacity of between 300 and 500 times greater than conventional concrete. In contrast to the conventional ordinary concrete, ECC display self-controlled crack width under increasing load. Usually, after initial loading, there is a formation of small numbers of cracks within the material which then spread. The widening progress until an average of about 60  $\mu\text{m}$  which is a product of ECC micro-mechanical tailoring [7]. Better strain capacity and multiple cracking are achieved in ECC through the use of fine sand only [8,9]. The elimination of coarse aggregates in ECC mixtures resulting in a higher content of cement in respective of conventional concrete structures. For instance, a typical ECC can contain cement that is more than 1000  $\text{kg}/\text{m}^3$ . It is established through studies, that each ton of cement produced give rise to an equal quantity of carbon dioxide, which is believed to be the origin of global greenhouse gas emission created by mankind, at least up to 5% [10]. Based on this fact, it is crucial to modify ECC mixture and develop a more environmentally friendly composite material by the incorporation of a natural mineral admixture to partially replace the total cement contents. Likewise, the current trends of ECC make use of Fly ash. It constitutes the necessary ingredients for the formation of ECC to improve the mechanical properties and as well reduce the shrinkage [11,12]. Nonetheless, delay of setting time and depression of early age mechanical strength as well as incompatibility with some other type of cement which may result in early stiffened of concrete materials limit the effectiveness of ECC in some special applications structurally. Hence, this research study takes in an effort to apply a natural fine aggregate with varied size ranges and a natural admixture, MK, produced in the laboratory from purified Kaolin as a partial replacement of cement. The developed HCC is better in early ages mechanical properties.

Natural pozzolans generally are included in concrete mixtures to aid in the conversion of calcium hydroxide (CH) which is separated as a less desirable hydration product into the more desirable calcium silicate hydrate (C-S-H). ASTM C 618 spells out class N porcelain as raw or calcined natural pozzolans and the frequently used natural pozzolans in the present dispensation are processed materials, it is treated with heat in a high temperature kiln and then grounded into a fine powdery form. They are metakaolin, calcined clay and calcined shale. Calcined clay is used in general construction like other pozzolans while calcined shale also has some cementing or hydraulic properties. Meanwhile, metakaolin is a product of calcination of high purity kaolin clay at a low-temperature and then grounded to an average particle size of about 1–2  $\mu\text{m}$ . This is considered to be about ten times finer than cement, but yet 10 times coarser in sizes compares to silica fume [13].

The incorporation of various supplementary cementing materials (SCM's) in concrete can result to significant effect on early age properties (both fresh and hardened). MK, for instance, was found to improve compressive and indirect tensile strengths, according to past studies, MK mixtures incorporation with between 5% and 10% MK by mass of cement [14]. In another related study, Li and Ding [15] investigated 10% cement weight replacement with MK, combine it with only OPC or with OPC and ultra-fine slag. The results revealed that the compressive strength of the mortar mixture with the incorporation of MK was found to be higher in value than the control mixtures and it was recorded to be approximately 8Mpa higher as at 28 days. Although, it was also recorded that the MK-slag mixtures manifested the highest 28-day strength. These strength contribution were to be assessed as a result of three basic factors which are; the effect of the filler, the OPC hydration

acceleration and the pozzolanic reaction of MK with the CH [16]. MK, also caused an increase in the autogenous shrinkage when measured from the age of 24 h according to past studies [17].

## 2. Objectives

This study aimed to investigate the characteristics of laboratory produced metakaolin (MK) and the early age mechanical strength properties of the hybrid cementitious composites produced by the replacement of binder weight by 10% MK, 1% colloidal nanosilica (CNS) and 1% epoxy resin without hardener. The main focus will be; first, to characterise the MK relatively to particle size distribution, specific surface area, mineralogical phases and chemical composition of the laboratory produced MK, and second, to investigate the early age mechanical strength of hybrid cementitious composites (HCC) with the incorporation of MK, CNS and epoxy. Lastly is to study the drying shrinkage properties of the HCC.

## 3. Material and methods

### 3.1. Materials

#### 3.1.1. Metakaolin

The MK used in this study was produced in the laboratory using the purified Kaolin supplied by a local supplier called Scancem materials Sdn. Bhd. The calcinations of the Kaolin to metakaolin was done using the ELE International laboratory muffle furnace at 800 °C for 1 h. After the calcinations, the MK was sieved using 150  $\mu\text{m}$  through the EFL 2000 Endecotts siever machine. The particle has 99% particles and its with a mean particle size diameter ( $d_{50}$ ) of 2.80  $\mu\text{m}$  with a specific gravity of 2.6. The specific surface area is 0.015  $\text{m}^2/\text{kg}$ . The chemical composition of the MK produced in the laboratory is shown in Table 1.

#### 3.1.2. Cement

The Ordinary Portland cement that complies with ASTM Type I Portland cement with a median particle size value of 6.14  $\mu\text{m}$ , the specific surface area of 1123.3  $\text{m}^2/\text{kg}$  and the specific gravity of 3.02 were used in this experimental study. The initial setting time and final setting time in minutes after the test are 115 and 310 respectively. The physical and chemical properties of the cement used is in accordance with the specifications in ASTM Standard C150 (ASTM, 1997b). The chemical composition of the OPC is shown in Table 2.

#### 3.1.3. Nanosilica

Colloidal nanosilica (CNS) was used in this research study as a cementitious material. The nanosilica used in this study was a product of Sigma-Aldrich product supplied locally. Specifically, it's Ludox AS-40 colloidal silica 40 wt.% suspension in water ( $\text{H}_2\text{O}$ ). The total weight is 60.08 g/mol. The physical and chemical properties are as stated in Table 3.

#### 3.1.4. Epoxy resin

This research study made use of epoxy resin (CP 370A) bisphenol A. The epoxy resin was used as cementitious material and as a bonding agent in the mixture. The physical composition of the epoxy is highlighted in Table 4.

**Table 1**

The physical properties of metakaolin produced in the laboratory in % weight.

$\text{SiO}_2$	$\text{TiO}_2$	$\text{Al}_2\text{O}_3$	$\text{Fe}_2\text{O}_3$	MnO	MgO	CaO	$\text{Na}_2\text{O}$	$\text{K}_2\text{O}$	$\text{P}_2\text{O}_5$
53.03	0.93	35.63	1.81	0.02	0.57	0.04	0.04	1.88	0.06

The LOI (Loss on ignition) = 1.99% obtained in compliance with BS EN 15935 (2009).

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