



## Ultrasonic dispersion of micro crystalline cellulose for developing cementitious composites with excellent strength and stiffness



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### ABSTRACT

This paper reports the first attempt to utilize ultrasonication energy for homogeneously dispersing microcrystalline cellulose (MCC) to develop MCC reinforced cementitious composites. Aqueous suspensions of MCC (1.0–5.0 wt.% with respect to water) were prepared using ultrasonication treatment and the suspensions were then added to the cement mortar mixes. The aqueous suspensions were characterized using optical microscopy for the area of MCC agglomerates and using UV–Vis spectroscopy for the concentration of well dispersed MCC and extractability and accordingly, the ultrasonication time was optimized. The developed cementitious composites, after 28 days of hydration, were characterized for their flexural and compressive properties. Selected samples were also analyzed for fracture surface, porosity and degree of cement hydration. Experimental results suggested that an ultrasonic treatment of 30 min could ensure good MCC dispersion with low agglomerated areas and high extractability. Flexural modulus, flexural strength and compressive strength improved strongly with MCC addition, leading to maximum improvements of 96%, 19.2%, and 51.4% using only 1 wt.% MCC. Moreover, addition of MCC to cementitious composites resulted in improved cement hydration and reduction in pore size of cementitious composites.

### 1. Introduction

In recent times, reinforcement of polymeric and cementitious materials by plant based fibres has attracted great attention. Natural fibres are considered as sustainable materials due to their renewability, environmental benefits and good specific mechanical properties (Rana et al., 2014). Reinforcement of polymeric and cementitious matrices using various plant fibres were found to improve the mechanical strength, stiffness and fracture toughness of composites and have been already applied in various industrial sectors including construction, automobiles, sports, etc. (Barra et al., 2015; Cho et al., 2013; Mármol et al., 2013; Soltan et al., 2017; Tingju et al., 2013). However, plant fibre based composites suffer from lower durability due to degradation of plant fibres as well as poor thermal and fire resistance. Poor interface with various matrices is another main concern while developing composites with plant fibre reinforcements. The most common approach to overcome these problems is through surface modification of plant fibres using various physical (such as plasma, corona, UV radiation, etc.) and chemical treatments (such as alkali, permanganate, silane, peroxide, ozone, etc. (Cho et al., 2013; Relvas et al., 2015; Tingju et al., 2013).

Another promising approach to enhance the properties and durability of plant based fibres is the deconstruction (commonly known as top-down approach) of plant cellulose into nano and micro fibrous materials such as nano fibrillated cellulose (NFC), nano crystalline cellulose (NCC), micro fibrillated cellulose (MFC) and micro crystalline cellulose (MCC) (Eichhorn et al., 2010; Parveen et al., 2017c; Peng et al., 2011). Bacterial cellulose (BC) is another type of cellulosic nanomaterial obtained from fermentation of low molecular weight sugars by bacteria (commonly known as bottom-up approach). As compared to plant based fibres, these nano and micro cellulosic materials present high mechanical properties, high surface area and interesting barrier and optical properties and are, therefore, considered as potential reinforcements for composite materials (Eichhorn et al., 2010; Parveen et al., 2017c; Peng et al., 2011). Nano and micro cellulose have been extensively studied in polymer matrix composites and have been applied in different industrial sectors including food, cosmetics, medical and hygiene products, emulsions and so on. (Eichhorn et al., 2010; Parveen et al., 2017c; Peng et al., 2011).

In order to reduce the carbon footprint, today's construction industries are constantly looking for new eco-friendly reinforcements of

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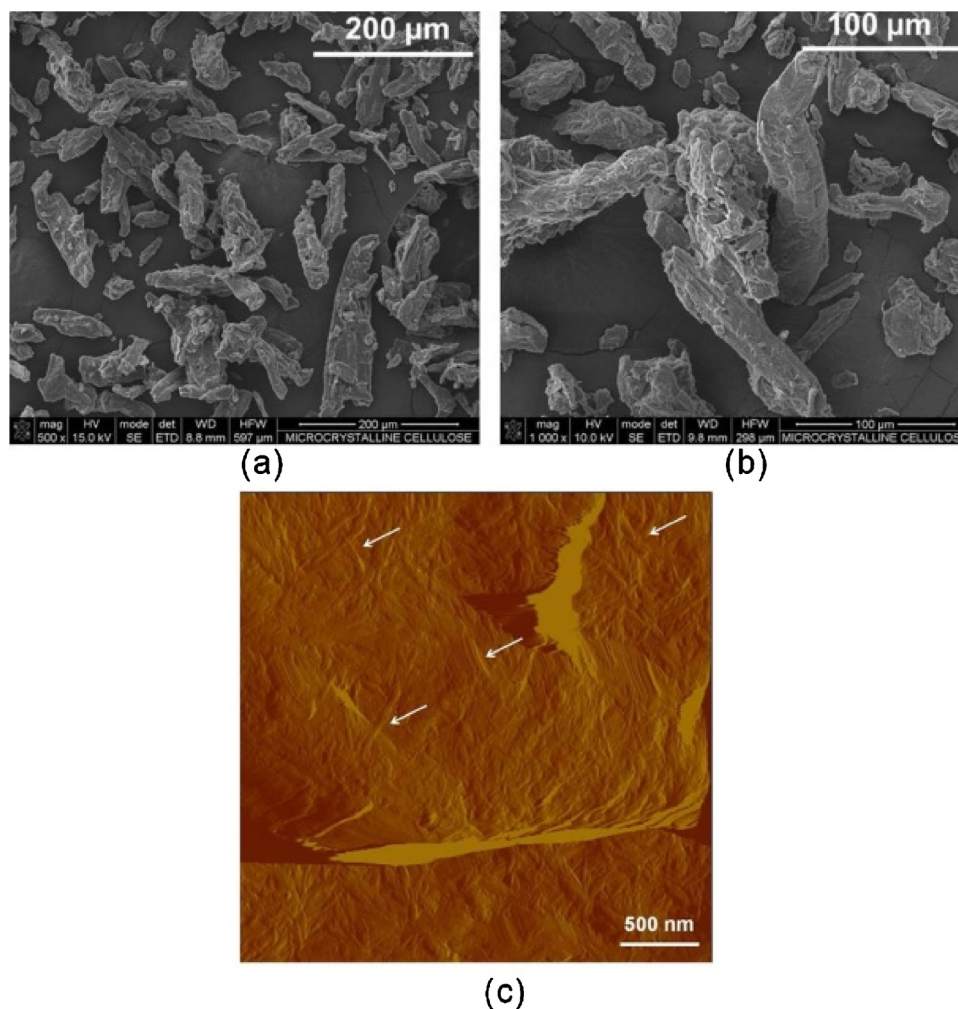


Fig. 1. SEM micrograph of MCC powder at different magnifications (a & b) and AFM image of MCC showing presence of NCC, as indicated by arrows (c).

cement based or polymer concretes for reducing the use of concrete and other metallic or synthetic reinforcements (such as steel, glass, carbon, etc.) (Brostow and Lobland, 2017; Martinez-Barrera et al., 2011). To replace these materials, various high performance nanostructures such as carbon nanotubes (CNTs) have been utilized to develop cementitious composites with considerable improvements in the microstructure and mechanical performance at very low CNT loading (Liew et al., 2016; Parveen et al., 2015; Parveen et al., 2013). However, difficulties in dispersion and high cost are very critical issues with these nanomaterials, which are yet to be solved for practical applications. Looking at the sustainability aspects, plant fibre (such as sisal, flax, hemp, bamboo, etc.) reinforced cementitious composites have been already studied to a considerable extent (Barra et al., 2015; Cho et al., 2013; Mármol et al., 2013; Soltan et al., 2017; Tingju et al., 2013). Currently, owing to the excellent properties of cellulose nano and micro materials, their use as the reinforcement of cementitious composites is getting increasing research attention. A very promising result was achieved by Cao et al. (2015) through the use of well dispersed NCC obtained from acid hydrolysis of plant cellulose. An improvement of flexural strength of cementitious composites up to 30% was obtained (Cao et al., 2015). A similar attempt was made by Mazlan et al. (2016) and achieved 42–45% improvement in compressive strength of cementitious composites by adding 0.2% NCC suspension obtained from acid hydrolysis. In another study, addition of NFC suspension, obtained by acid hydrolysis, to oil well cement (OWC) resulted in significant improvement in the yield stress of OWC slurry, degree of hydration and flexural strength (by 20.7% using 0.04 wt.% of NFC) of cement composites (Sun

et al., 2017). Jiao et al. (2016), on the other hand, used TEMPO oxidized NFC suspensions to reinforce cementitious composites and achieved 15% and 20% improvements in flexural and compressive strengths, respectively using 0.15 wt.% NFC. Also, the use of BC surface coating on bagasse fibres was found to significantly improve the fibre/cement interface and reduce the fibre mineralization (Kazemi et al., 2015). However, NCC and BC are not commercially available and have to be prepared prior to use in cementitious composites. MCC, on the other hand, is commercially available in powder form and therefore, practically more suitable for use in the current construction industries.

In contrast to nano cellulose, research on MCC based cementitious composites is very rare in the existing literature. Recently, Hoyos et al. (2013) used commercially available MCC powder, saturated with water and then added to the cement mixture to fabricate cementitious composites. This process, however, could not ensure homogeneous MCC dispersion in aqueous medium as well as in cementitious matrix and consequently, no improvement in mechanical performance was achieved (Hoyos et al., 2013). In another study, surface modified MCC with tetraethyl orthosilicate (TEOS) was added to cement composites, resulting in strong improvement (~60% improvement in flexural strength after 28 days and 45% in compressive strength) of mechanical properties of cementitious composites (Anju et al., 2016). Frequently, nano and micro cellulosic materials are chemically functionalized with different functional groups such as carboxylic, silane, acetyl, ester, polymers, etc. to improve their compatibility with different matrices (Berlioz et al., 2009; Cao et al., 2009; Kaboorani and Riedl, 2015; Salajková et al., 2012). Also, MCC dispersion can be improved

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