# PERFORMANCE OF CEMENT MORTAR CONTAINING CELLULOSIC NANOCRYSTALS FROM PALM OIL WASTE

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A thesis submitted in fulfilment of the requirements for the award of the degree of Doctor of Philosophy (Civil Engineering)

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## **DEDICATION**

Dedicated to the memory of my father, Mazlan Abu Mansor and my mother, Siti Aisah Abdul Wahid who always believed in my ability to be successful in the academic arena.

You are gone but your belief in me has made this journey possible.

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#### ABSTRACT

A study on the utilization of cellulose nanocrystals (CNCs) as a natural-base additive in cement mortar as a strengthening agent had been conducted. Since cementbased materials such as concrete and mortar are known as the most utilized materials on earth after water, various studies on the cement mortar have been done to improve its properties to make more compatible with the current demand. Nowadays, the development of construction industries is going towards producing cement mortar that high in strength and environmentally friendly. However, to develop a cement mortar that as strong as concrete is challenging due to no coarse aggregate is used in the mortar mix. Thus, the development of new technologies and materials that can improve the strength of mortar without the usage of coarse aggregates was studied. These days, additive to strengthen the cement mortar by using natural resources had gained interest among researchers. Still, limited study had been conducted to study the outcome of natural-based additive in cement mortar as a strengthening agent. Thus, this research aims to investigate the changes in cement mortar properties and behavior after it is affected by a natural-based additive known as cellulose nanocrystals (CNCs). CNCs, known as advanced materials produced by using current advanced nanotechnologies and bring out the strongest part of the cellulose which is stronger than a strength of steel in tensile. The CNCs was used in cement mortar to study deeply on its characteristic and improvement contributed by CNCs. In this study, the mortar specimens were prepared with a mass ratio of 1: 2.75 (cement: fine aggregates), the water-cement ratio of 0.47 and 0.2 to 0.8% of CNCs addition by weight of cement content. Three types of curing regimes were executed known as a wrap, lime and water curing in order to find the most compatible curing method with CNCs mortar. The wrap curing method was found to produce better strength performance with the addition of CNCs compared to lime and water curing during the pilot study. Therefore, wrap curing was used throughout the study. The normal mortar was prepared as a control sample. Three important phases of tests were carried out in this study starting from chemical and physical tests characterization of the CNCs. This was followed by the performance of fresh and hardened properties of cement composites after incorporating the CNCs and ended with thermal performance tests of the CNCs mortar. The experimental results showed that the optimum percentage of CNCs used in the mix was 0.4% as it resulted in higher compressive, flexural and tensile strength. The cement mortar containing CNCs was found to perform effectively in improving the strength properties up to 43.6 %, 20 % and 147 % for compressive, flexural and tensile strength, respectively, as well as alter the thermal conductivity for 0.1% and 0.2% for the thermal resistance of cement mortar. All tests were carried out by following the American Standard Testing Method (ASTM), British Standards (BS) and Japanese International Standards (JIS) standards. Overall, test results including microstructure studies showed that CNCs can be used as an additive in cement mortar to strengthen the cement properties.

#### ABSTRAK

Kajian penggunaan nanokristal selulosa (CNCs) sebagai bahan tambahan semulaiadi dalam mortar simen sebagai agen penguat telah dijalankan. Memandangkan bahan berasaskan simen seperti konkrit dan mortar dikenal pasti sebagai bahan yang kerap digunakan di bumi selepas air, pelbagai kajian terhadap mortar simen telah dijalankan bagi meningkatkan sifatnya untuk menjadikannya lebih sesuai dengan permintaan semasa. Pada masa kini, perkembangan industri pembinaan sedang menuju kearah penghasilan simen mortar berkekuatan tinggi dan mesra alam. Walau bagaimanapun, penghasilan mortar simen berkekuatan tinggi seperti konkrit adalah mencabar memandangkan agregat kasar tidak digunakan dalam campuran mortar. Sehubungan itu, pembangunan teknologi baru dan bahan bagi menambah baik kekuatan mortar tanpa penggunaan agregat kasar telah dikaji. Kebelakangan ini, pengunaan bahan tambah bagi meningkatkan kekuatan mortar simen dengan menggunakan bahan semulajadi telah menarik perhatian para penyelidik. Namun, kajian yang telah dijalankan bagi mengkaji kesan bahan tambahan semulajadi sebagai agen penguat dalam mortar simen adalah terhad. Oleh itu, tujuan kajian ini dijalankan adalah untuk mengkaji perubahan kepada sifat mortar simen dan kelakuannya selepas didedahkan kepada bahan tambah semulajadi yang dikenali sebagai nanokristal selulosa (CNCs). CNCs yang juga dikenali sebagai bahan terkini yang dihasilkan menggunakan teknologi nano terkini, mengeluarkan bahagian terkuat selulosa yang mana ianya lebih kuat berbanding kekuatan keluli dalam tegangan. CNCs digunakan dalam campuran mortar bagi mengkaji dengan lebih mendalam sifat dan penambah baikan yang disumbangkan oleh CNCs. Dalam kajian ini, spesimen mortar disediakan dengan nisbah berat 1: 2.75 (simen : agregat halus), nisbah air-simen 0.47 dan tambahan CNCs sebanyak 0.2 hingga 0.8% berdasarkan berat simen. Tiga kaedah pengawetan telah dijalankan iaitu pembungkusan, kapur dan pengawetan air bagi mengenalpasti kaedah yang paling sesuai bagi mortar CNCs. Kajian mendapati kaedah pengawetan secara pembungkusan menghasilkan prestasi kekuatan yang lebih baik bagi tambahan CNCs berbanding pengawetan kapur dan air semasa kajian perintis dilaksanakan. Oleh itu, kaedah pengawetan secara pembungkusan digunakan sepanjang kajian dijalankan. Mortar biasa disediakan sebagai sampel kawalan. Tiga fasa penting ujikaji telah dijalankan bermula dengan ujian kimia dan ujian fizikal CNCs. Ujikaji diteruskan dengan ujian prestasi konkrit basah dan konkrit keras komposit simen selepas menggabungkan CNCs dan berakhir dengan ujian prestasi termal mortar CNCs. Hasil kajian mendapati CNCs berkesan secara efektif dalam meningkatkan sifat kekuatan sehingga 43.6%, 20% dan 147% bagi kekuatan mampatan, lenturan dan tegangan disamping mengubah kekonduksian terma untuk 0.1% dan 0.2% bagi rintangan terma simen mortar. Ujian-ujian yang telah dijalankan adalah berdasarkan kepada American Standard Testing Method (ASTM), British Standard (BS) dan Japanese International Standards (JIS). Hasil kajian secara menyeluruh termasuk kajian terhadap mikrostruktur mendapati CNCs boleh digunakan sebagai bahan tambah dalam mortar simen untuk meningkatkan sifat simen.

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### LIST OF ABBREVIATIONS

CNCs	-	Cellulose nanocrystals
MCCs	-	Microcrystalline cellulose
OPC	-	Ordinary Portland Cement
XRF	-	X-ray Fluorescence
TEM	-	Transmission Electron Microscopy
ASAP	-	Surface Area and Porosity Analyser
XRD	-	X-ray Diffraction
NDT	-	Non Destructive Test
FESEM	-	Field Emission Scanning Electron Microscopy
SEM/EDX	-	Scanning Electron Microscopy /Energy Dispersive X-ray
MLA	-	Mineral Liberation Analysis
FTIR	-	Fourier Transform Infrared Spectroscopy
TG/DTA	-	Thermo-gravimetric and Differential Temperature Analysis
ASTM	-	American Standard and Testing Manual
UPV	-	Ultrasonic Pulse Velocity
BS	-	British standard
HFM	-	Heat Flow Meter
LSP	-	Lignosulfonate plastisizer
NFS	-	Formaldehyde superplastisizer
ASTM	-	American Standard Testing Method
BS	-	British Standard
JIS	-	Japanese International Standard
TEMPO	-	Tetramethylpiperidine-1-oxyl
NDF	-	Neutral Detergent Fibre
ADL	-	Acid Detergent Fibre
ADF	-	Acid Detergent Lignin
MPOB	-	Malaysia Palm Oil Board
BET	-	Brunauer-Emmet-Teller
HFM	-	Heat Flow Meter
FLIR	-	Forward Looking Infrared Camera

- ISO International Organization for Standardization
- RH Relative humidity
- UHI Urban Heat Island

## LIST OF SYMBOLS

°C	-	Celsius
W/W	-	Mass fraction
v/v	-	Volume concentration
m/v	-	Mass concentration
w/c	-	Water/cement ratio
L/D	-	Aspect ratio
kV	-	Kilo volt
%	-	Percent
L	-	Length
b	-	width
d	-	Thickness
Р	-	Load
А	-	Area
σc	-	Compressive strength
GPa	-	Giga Pascal
cp	-	Centipoise
π	-	Pie
Н	-	Height
Cu K-a	-	Copper K alpha
α	-	Alpha
0	-	Degree
P/Po	-	Relative Pressure
RH	-	Relative Humidity
<b>O</b> 3	-	Silica
Al 2 O 3	-	Alumina,
Fe 2 O 3	-	Iron oxide
CaO	-	Calcium Oxide
MgO	-	Magnesia
$N_2O$	-	Sodium

P 2 O 5	-	Phosphorus
CaCO <sub>3</sub>	-	Carbonate
$Ca(OH)_2$	-	Calcium hydroxide
Ta	-	Ambient temperature
CaSiO <sub>5</sub>	-	Alite
Ca <sub>2</sub> SiO <sub>4</sub>	-	Belite
$CO_2$	-	Carbon dioxide
mm	-	Millimetre
μm	-	Micrometer
φ	-	Diameter
β	-	Beta
nm	-	Nanometer
Ν	-	Normality
g	-	Gram
mV	-	Millivolt
Hg	-	Mercury
kN	-	Kilonewton
CSH	-	Calcium Silicate Hydrate
Ca(SO) <sub>4</sub>	-	Calcium Sulphate
SiO <sub>2</sub>	-	Silicon Oxide
CaSiO <sub>3</sub>	-	Calcium Silicon Oxide
MPa	-	Mega Pascal
θ	-	theta
kBr	-	Potassium Bromide
psi	-	Pound per square inch
$WK^{-1}m^{-1}$	-	Watt per kelvin-meter
W/m <sup>2</sup>	-	Watt per meter square

#### **CHAPTER 1**

#### INTRODUCTION

#### **1.1 Background of the study**

The utilization of wastes products in construction materials has been widely investigated and studied by numerous researchers across the world (Johari et al., 2014). Malaysia is known as a country that produces high biomass production annually. Being a major agricultural commodity producer in the region, Malaysia produces at least 168 million tonnes of biomass in 2018, including timber and oil palm waste, rice husks, coconut trunk fibres, municipal waste and sugarcane waste annually (Hamzah et al., 2019). Most of these wastes were left rot at the field without commercial return (Abdullah and Sulaiman, 2013). This resulting in increasing the cause of air pollution due to open burning.

Agricultural waste knows with its higher lignocellulose content which is known as the most abundant and renewable natural biopolymer on earth. Cellulose can be found in plants such as flowers, trees, weeds, grasses, vines, and bushes. Other living things such as animals (mammals, herbivore, carnivore and omnivore) and bacteria like Acetobacter xylinum, Acetobacter, Azotobacter, Rhizobium, Pseudomonas, Salmonella, Alcaligenes etc. also contain a significant amount of cellulose (Abdul Khalil et al., 2012, 2014; Abdullah and Sulaiman, 2013). Cellulose fibres or used as reinforcing materials in cement mortar materials help to improve the performance of mortar by increasing the resistance towards tensile stress, shrinkage, and modulus (Mohammed et al., 2015). For that reason, the studied on the cellulose fibres in cement composites materials have been studied comprehensively in many ways by using various types of cellulose fibres (Sajjala, 2018a). However, drawbacks from the direct usage of cellulose in cement materials such as workability and degradation of strength after a certain period cause a new study on surface modification on cellulose (Mohammed et al., 2015). Therefore, with the advanced

technology, the surface-modified cellulose knows as cellulose nanocrystals (CNCs) was introduced and widely used in many industries such as automotive, aerospace, electronics, pharmaceutical and food industries (Barra *et al.*, 2015; Soltan *et al.*, 2017).

CNCs was discovered as early as 1949, however, CNCs did not receive much attention because of their nano sizes that cannot be imaged under present optical and microscopy equipment during that era (Eyley and Thielemans, 2014). Then, in 1992, the interest in CNCs was revealed when their colloidal suspensions unexpectedly showed liquid crystalline properties which been found when the advent of higher resolution imaging techniques such as atomic force microscopy (AFM) and transmission electron microscopy (TEM) (George and Sabapathi, 2015). The immense strength and stiffness of cellulose crystallites soon spurred interest in their potential to reinforce polymers.

These good properties of CNCs can be achieved by appropriate modification of CNCs in terms of physical, chemical or rheological properties based on desired improvements. The nanoparticles of CNCs are stabilized in aqueous suspension during the hydrolysis process with the form of negative charges on the surface of the nanoparticles (Pelissari *et al.*, 2014; Tian *et al.*, 2016). Furthermore, CNCs nanoscale particles are promising can be used as reinforcing agents in composites to produce high-performance nanocomposites. This phenomenon is due to the CNCs' basic physicochemical properties which content an anisotropic chiral nematic liquid crystalline phase (Eyley and Thielemans, 2014).

Cement mortar is a strong and relatively cheap construction material and is, therefore, present the most widely used material in the construction industry. The main constituent that contributes to mortar strength is Portland cement. It is estimated that cement (Portland clinker) production alone contributes about 7% of the global carbon dioxide emissions due to the burning of limestone and clay at a temperature of 1500 °C. During this process, calcium carbonate (CaCO<sub>3</sub>) is converted to calcium oxide (CaO) and carbon dioxide is released (McDonald *et al.*, 2019). From an environmental standpoint, cement does not appear to be a sustainable material (Bediako *et al.*, 2016).

Therefore, the usage of a non-sustainable additive such as superplasticizer to improve the workability should be reduced.

Nowadays, the production of cement mortar that satisfied the application of lightweight industries such as in high rise buildings and floating structures is very hard to achieve (Takumi *et al.*, 2009). In lightweight industries, the strength and density of the composites were the major criteria that been observed. However, it very hard to achieve both high strength and low density at the same time by using the conventional method. This due to the basic theory of the cement composites strength is proportional to the density (Neville, 2006). Therefore, various options were introduced in the previous studies by using chemical or natural-based additive or cement-based replacement such as resin, fly ash, bottom ash, steel fibre and many more (Mohammed *et al.*, 2015; Khalid *et al.*, 2016; Zhang *et al.*, 2019)

In construction industries nowadays, various studies on developing better construction materials such as high strength cement composites or environmental friendly composites, which can be used in skyscrapers, bridges, sidewalks, highways, houses, and dams (Martínez-Barrera *et al.*, 2014). Besides that, in the production of cement mortar, the existing conventional mortar is known as a non-load bearing structure. This is due to the cement mortar matrix that does not contain coarse aggregates like a concrete matrix. Therefore, the addition of additive or reinforcing materials were needed to improve the performance of the mortar. Hence, causing difficulties in workability, dispersion of fibre, and reinforcing costs.

Based on the achievement of previous studies in discovered the unique properties of CNCs in improving various composites materials in diverges industries. Hence, this study discussed the effect of CNCs extracted from palm oil waste incorporated in cement mortar in enhancing its performance in terms of strength, microstructural and thermal performance.

#### **1.2 Problem statement**

Nowadays, the production of waste from palm oil industry has been increased from 13.9 million tonnes in September 2018 to 15.9 million tonnes in September 2019 (Malaysian Palm Oil Board, 2019). Demand in new construction building materials, such as strong, lightweight, thermally efficient, environmentally friendly and low-cost materials have been extremely high. However, to produce this type of material need advanced technology. These advanced technologies will cost a high amount of money.

Nowadays production of high strength cement mortar needs various addition of costly admixture to improve the quality of the physical performance of the cement composites (Mehra *et al.*, 2016; Zhang *et al.*, 2019). Example of mineral admixture and chemical admixture that been used in the cement composites industries to produce the high strength concrete or mortar such as fly ash or silica fume and naphthalene formaldehyde superplasticizers (NFS) or lignosulfonate plasticizers (LSP), respectively. Therefore, eco-friendly and low-cost admixture should be introduced and studied, to reduce the usage of mineral and chemical admixture in the later future.

Moreover, there are so many research has been done by the addition of natural or synthetic fibres in cement mortar or concrete to strengthen the composites. In Malaysia, the most common natural fibres that been used in the cement mortar as fibre reinforced material mix, were pineapple fibre, palm oil fibre, kenaf fibre, paddy straw fibre, sugarcane fibre, and banana fibre (Mohammed *et al.*, 2015; Sajjala, 2018a). However, this effort by using the micro sizes of fibres in the cement mortar mix cause difficulty and limitation in the workability stage. The higher the addition of micro sizes fibre will reduce the workability of the cement mortar as balling effect occurred during mixing. Hence, reduce the strength of the cement mortar as the aftermath.

On top of that, the addition of admixture or natural fibres in cement mortar provides any guarantee for long-lasting strength of the structure. This due to the natural behaviour of the micro sizes fibre, which exposed to decay after a long haul (Ahmad *et al.*, 2019). This decay effect will speed up if the cement mortar is not dense enough which provide more void or passage for air or water to penetrate inside the structure.

Therefore, an early precaution of the well-protected structure of cement mortar to prevent chemical attack and decay agents such as air or water to penetrate.

Thermal performance of cement mortar is important properties that need to be studies nowadays (Shahedan *et al.*, 2017). The extreme changed in the climate across the globe causing the importance of producing the construction material that has good thermal insulation rather than just toughness. The utilization thermal insulation of building becomes a great potential to reduce the building thermal load and consequently its energy consumption especially in harsh weather and lead to improvement of economical aspects of buildings. Therefore, more comfort in living and significant energy saving can be realized by buildings with proper materials, design and operation.

### 1.3 Aims of the study

Many studies in construction have expanding and focusing their studies on producing green and sustainable construction materials. Despite this strategic direction, the literature reveals that relatively little is known about the performance cement mortar incorporating with cellulose-based materials. Consequently, this research aims to narrow this research gap and conduct the research on the performance of CNCs incorporated in cement mortar, focusing on its physical and thermal performance. The results will be used to provide recommendations to the construction industries on the potential usage of CNCs mortar as a green and sustainable construction material.

#### **1.4 Objectives of the study**

Several objectives can be drawn according to the problem statement. The objectives are as follow:

- 1. To characterize the physical and chemical properties of cellulose nanocrystals (CNCs) extracted from palm oil fruit bunches wastes.
- 2. To determine the engineering properties of CNCs mortar by using the optimum amount of CNCs content.
- 3. To identify the contribution of CNCs in improving the mortar performance through microstructure study.
- 4. To investigate the performance of CNCs addition in cement mortar in terms of its impact towards heat

## 1.5 Research question

Four major questions need to be answered from this research:

- 1. What are the crucial components of CNCs that make it suitable to improve mortar physical and thermal performance?
- 2. How the addition of cellulose nanocrystals can contribute to the strength of the mortar?
- 3. What are the changes in microstructure study of mortar before and after incorporating CNCs?
- 4. How does the CNCs affect the mortar properties in improving the thermal performance?

#### **1.6** Scope and limitation of the study

The versatility in the formulation of cellulose mortar led to wide acceptance in the construction industry. CNCs is potential to be used as alternative materials other than know cementitious materials such as palm oil fuel ash, slag or rice husk. This research focused on assessing the effect of CNCs addition in mortar matrix based on strength development, microstructures study and thermal performance. To achieve the expectation of the research, a limitation has been set up for this project by following the standard specification:

- (a) Percentages of CNC as an additive are tested with 0.2 to 0.8% with0.2% increment by volume of cement.
- (b) The sizes of sand passing sieve between 850 μm to 600 μm and 600μm to 150μm.
- (c) Test samples are including 50mm cubes for compressive strength,  $40 \times 40 \times 160$  mm prism for flexural strength test,  $\Phi 70 \times 150$  mm cylinders for splitting tensile test and 300x300x100mm bricks for thermal behaviour tests.
- (d) Three types of curing regimes namely wrap, water, lime was used.

The tests were conducted based on the American Standard Testing Method (ASTM) standard and British Standard (BS). Some testing methods were adopted from previous researchers since it was not stated in any other established standard.

#### **1.7** Significance of the study

Various application of CNCs reported by in previous studies by using traditional and advanced technology have unveiled many potentials of CNCs. Several studies found that, different sources of cellulose resulting in different properties of CNCs. In this study, the sources of cellulose which came from the abundant local agricultural waste which is palm oil fruit bunch waste have been chosen as the source of cellulose. With the usage of palm oil fruit bunch waste, the carbon emission from the palm oil industries can be reduced to a certain level. This due to less open burning activities will occur in order to dispose the waste.

The overall study can be summarised that the properties of cement mortar can be enhanced by using natural based additives. The natural additives such as CNCs can be a great opportunity to improve the physical properties, inner structure, mechanical properties and thermal performance in a sustainable and green way. Thus, with the improvement in the properties of cement mortar, the mortar can be applied in varies application such as good insulator for building, high rise building and bridge construction material.

With the positive effect of CNCs addition in cement mortar, this can be used to improve the conventional cement mortar used in the construction industries. With a little addition of CNCs can improve the properties of cement mortar. Since the CNCs is a nanomaterial, the little addition of the CNCs would not change the appearance of the common mortar. It only improves the inner structure of the cement mortar.

#### **1.8** Thesis outline

Chapter 1 (Introduction): Chapter 1 discusses the background of the research comprising the statement of the problem, research objectives, research scope, the significance of the research, and limitations of the study.

Chapter 2 (Literature Review): Chapter 2 initially discusses the past research from various researchers all around the world. The main focus is to identify important performance criteria and parameters research that had been done and compared with current research. This chapter then discusses the history, studies, science and different approaches to the utilization of CNCs and its ability to enhance the properties of materials. The differing performance of CNCs as admixture evaluation methods are discussed by looking at the different admixture that each researcher had reported. Chapter 3 (Research Methodology): Chapter 3 focuses on a testing method that had been conducted to evaluate the performance of CNCs-mortar. The methodology is separated into three parts: a) the characterization of CNCs as potential additive of cement composites, b) hardened properties of mortar incorporating CNCs, and c) microstructure observation of the CNCs-mortar. All the testing methods and parameters are discussed in this chapter.

Chapter 4 (Characterizations of Raw Materials of Cellulose Nanocrystals): Chapter 4 reports and explains the analyses undertaken for achieving the first objectives of this research. The results are discussed in this chapter. Some sections from objective 4 based on microstructure morphology are also included.

Chapter 5 (Properties of Mortar Contain CNCs as Potential Admixture): Chapter 5 discusses the performance of CNCs-mortar as to achieve the Objectives 2, 3 and 4. The correlation analysis was carried out to evaluate CNCs as the potential cement composites admixture.

Chapter 6 (Thermal Behaviour of CNCs-mortar): Chapter 6 converse about the potential of CNCs in improving the thermal behaviour of the cement composites materials by incorporating it. This chapter explains the results and analysis made to satisfied Objectives 5.

Chapter 7 (Conclusions and Recommendations): Chapter 7 concludes the findings of the overall research work that was undertaken. The contribution and implications of the findings toward the construction industry in general and performance evaluation, in particular, are explained. The limitations and possible improvements for future undertakings are also discussed. Suggestions for future research are conveyed in the final part of this chapter.

#### REFERENCES

- Abdul Khalil, H. P. S., Bhat, a. H. and Ireana Yusra, a. F. (2012) 'Green composites from sustainable cellulose nanofibrils: A review', *Carbohydrate Polymers*. Elsevier Ltd., 87(2), pp. 963–979.
- Abdul Khalil, H. P. S., Davoudpour, Y., Islam, M. N., Mustapha, A., Sudesh, K., Dungani, R. and Jawaid, M. (2014) 'Production and modification of nanofibrillated cellulose using various mechanical processes: a review.', *Carbohydrate polymers*. Elsevier Ltd., 99, pp. 649–65.
- Abdullah, N. and Sulaiman, F. (2013) 'The Oil Palm Wastes in Malaysia', in *INTECH*, pp. 75–98.
- Abitbol, T., Rivkin, A., Cao, Y., Nevo, Y., Abraham, E., Ben-Shalom, T., Lapidot, S. and Shoseyov, O. (2016) 'Nanocellulose, a tiny fiber with huge applications', *Current Opinion in Biotechnology*. Elsevier Ltd, 39(I), pp. 76–88.
- ACI212.3R-91 (1993) 'Chemical Admixtures for Concrete', *Concrete International*, 15(10).
- Ahmad, R., Hamid, R. and Osman, S. A. (2019) 'Physical and Chemical Modifications of Plant Fibres for Reinforcement in Cementitious Composites', 2019.
- Al-Homoud, M. S. (2005) 'Performance characteristics and practical applications of common building thermal insulation materials', *Building and Environment*, 40(3), pp. 353–366.
- Alawadhi, E. M. (2015) The design, properties, and performance of concrete masonry blocks with phase change materials, Eco-efficient Masonry Bricks and Blocks: Design, Properties and Durability. Elsevier Ltd.
- Aliabdo, A. A., Elmoaty, A., Elmoaty, M. A. and Salem, H. A. (2016) 'Effect of water addition, plasticizer and alkaline solution constitution on fly ash based geopolymer concrete performance', *Construction and Building Materials*. Elsevier Ltd, 121, pp. 694–703.
- Almeida, A. E. F. S., Tonoli, G. H. D., Santos, S. F. and Savastano, H. (2013) 'Improved durability of vegetable fiber reinforced cement composite subject to accelerated carbonation at early age', *Cement and Concrete Composites*. Elsevier Ltd, 42, pp. 49–58.

- Anbalagana, G., Prabakaranb, A. R. and Gunasekaran, S. (2010) 'SPECTROSCOPIC CHARACTERIZATION OF INDIAN STANDARD SAND', *Journal of Applied Spectroscopy*, 77(1), pp. 124–128.
- Araki, J., Wada, M., Kuga, S. and Okano, T. (1998) 'Flow properties of microcrystalline cellulose suspension prepared by acid treatment of native cellulose', *Colloids and Surfaces A: Physicochemical and Engineering Aspects.*
- Arsene, M.-A., Bilba, K., Savastano Junior, H. and Ghavami, K. (2013) 'Treatments of non-wood plant fibres used as reinforcement in composite materials', *Materials Research*, 16(4), pp. 903–923.
- ASTM C128 (2015) 'Standard Test Method for Relative Density (Specific Gravity) and Absorption of Fine Aggregate', *American Society of Testing Materials*.
- ASTM C496/C496M (2017) 'Standard Test Methods for splitting tensile strength of cylindrical concrete specimens', *American Society of Testing Materials*.
- ASTM C518 (2017) 'Standard test method for steady-state thermal transmission properties by means of the heat flow meter apparatus', *American Society of Testing Materials*.
- ASTM C597-09 (2009) 'Pulse Velocity Through Concrete', American Society of Testing Materials, (Note 2), pp. 6–9.
- ASTMC109/C109M (2002) 'Standard Test Method for Compressive Strength of Hydraulic Cement Mortars (Using 2-in. or [50-mm] Cube Specimens)', *American Society of Testing Materials*, pp. 1–10.
- ASTMC230/C230M-14 (2010) 'Standard Specification for Flow Table for Use in Tests of Hydraulic Cement 1', *Annual Book of ASTM Standards*, pp. 4–9.
- ASTMC778-13 (2014) 'Standard Specification for Standard Sand', *American Society* of Testing Materials, pp. 1–3.
- Atalla, R. H. and Vanderhart, D. L. (1983) 'Native Cellulose: A Composite of Two Distinct- Crystalline Forms', *Cellulose*, 177, pp. 6–8.
- Bai, W., Holbery, J. and Li, K. (2009) 'A technique for production of nanocrystalline cellulose with a narrow size distribution', *Cellulose*.
- Barbhuiya, S. and Chow, P. L. (2017) 'Nanoscaled mechanical properties of cement composites reinforced with carbon nanofibers', *Materials*, 10(6).
- Barra, B. N., Santos, S. F., Bergo, P. V. A., Jr, C. A., Ghavami, K. and Jr, H. S. (2015) 'Residual sisal fibers treated by methane cold plasma discharge for potential

application in cement based material', *Industrial Crops & Products*. Elsevier B.V., 77, pp. 691–702.

- Beck-Candanedo, S., Roman, M. and Gray, D. G. (2005) 'Effect of reaction conditions on the properties and behavior of wood cellulose nanocrystal suspensions.', *Biomacromolecules*, 6(2), pp. 1048–54.
- Beck, S. and Bouchard, J. (2014) 'Effect of storage conditions on cellulose nanocrystal stability', *Tappi Journal.*, 13, pp. 53–61.
- Bediako, M., Adobor, C. Dela, Amankwah, E. O., Nyako, K. and Kankam, C. K. (2016) 'Maximizing the Sustainability of Cement Utilization in Building Projects through the Use of Greener Materials', 2016.
- Bera, A., Kumar, T., Ojha, K. and Mandal, A. (2013) 'Adsorption of surfactants on sand surface in enhanced oil recovery: Isotherms, kinetics and thermodynamic studies', *Applied Surface Science*.
- Bhutta, M. A. R. and Ohama, Y. (2010) 'Recent Status of Research and Development of Concrete-Polymer Composites in Japan', *Concrete Research Letters*.
- Bilba, K. and Antilles, U. (2003) 'Sugar cane bagasse fibre reinforced cement composites', (January).
- Bilba, K., Arsene, M. A. and Ouensanga, A. (2003) 'Sugar cane bagasse fibre reinforced cement composites. Part I. Influence of the botanical components of bagasse on the setting of bagasse/cement composite', *Cement and Concrete Composites*, 25(1), pp. 91–96.
- Bischof, R. H., Ramoni, J. and Seiboth, B. (2016) 'Cellulases and beyond : the first 70 years of the enzyme producer Trichoderma reesei', *Microbial Cell Factories*. BioMed Central, pp. 1–13.
- Bondeson, D., Mathew, A. and Oksman, K. (2006) 'Optimization of the isolation of nanocrystals from microcrystalline cellulose by acid hydrolysis', *Cellulose*, 13, pp. 171–180.
- Bondeson, D. and Oksman, K. (2007) 'Polylactic acid/cellulose whisker nanocomposites modified by polyvinyl alcohol', *Composites Part A: Applied Science and Manufacturing*.
- Brinchi, L., Cotana, F., Fortunati, E. and Kenny, J. M. (2013) 'Production of nanocrystalline cellulose from lignocellulosic biomass: technology and applications.', *Carbohydrate polymers*. Elsevier Ltd., 94(1), pp. 154–69.
- Brown, R. M. (1987) 'The biosynthesis of cellulose', Topics in Catalysis.

- Cao, Y., Zavaterri, P., Youngblood, J., Moon, R. and Weiss, J. (2015) 'The influence of cellulose nanocrystal additions on the performance of cement paste', *Cement* and Concrete Composites. Elsevier Ltd, 56, pp. 73–83.
- Cao, Y., Zavattieri, P., Youngblood, J., Moon, R. and Weiss, J. (2016) 'The relationship between cellulose nanocrystal dispersion and strength', *Construction and Building Materials*. Elsevier Ltd, 119, pp. 71–79.
- Chen, Y. W. and Lee, H. V. (2018) 'Revalorization of Selected Municipal Solid Wastes as New Precursors of "Green" Nanocellulose via a Novel One-Pot Isolation System: A Source Perspective', *International Journal of Biological Macromolecules*. Elsevier B.V., 107, pp. 78–92.
- Chen, Y. W., Lee, H. V., Juan, J. C. and Phang, S.-M. (2016) 'Production of new cellulose nanomaterial from red algae marine biomass Gelidium elegans', *Carbohydrate Polymers*. Elsevier Ltd.
- Cheng, F., Liu, C., Wei, X., Yan, T., Li, H., He, J. and Huang, Y. (2017) 'Preparation and Characterization of 2,2,6,6-Tetramethylpiperidine-1- oxyl (TEMPO)-Oxidized Cellulose Nanocrystal/Alginate Biodegradable Composite Dressing for Hemostasis Applications', *Sustainable Chemical Engineering*, 5, pp. 3819– 3828.
- Cheng, Q., Devallance, D., Wang, J. and Wang, S. (2006) 'Advanced Cellulosic Nanocomposite Materials'.
- Chirayil, C. J., Mathew, L. and Thomas, S. (2014) 'Review of recent research in nano cellulose preparation from different lignocellulosic fibers', *Reviews on Advanced Materials Science*, 37(1–2), pp. 20–28.
- CIDB (2015) 'The Construction Industry Transformation Programme', p. 2015.
- Claude Goguen (2012) 'Air Entrainment versus Air Entrapment', National Precast Concrete Association.
- Crow, J. M. (2008) 'The concrete conundrum', Chemistry World, (March), pp. 62-66.
- Csiszar, E., Kalic, P., Kobol, A. and Ferreira, E. D. P. (2016) 'Ultrasonics Sonochemistry The effect of low frequency ultrasound on the production and properties of nanocrystalline cellulose suspensions and films', ULTRASONICS SONOCHEMISTRY. Elsevier B.V., 31, pp. 473–480.
- Djalal, T., Hazwan, H. M. and Kumar, T. V. (2017) 'Recent progress in cellulose nanocrystals: sources and production', *Nanoscale*, pp. 17–25.

- Dole, P., Joly, C., Espuche, E., Alric, I. and Gontard, N. (2004) 'Gas transport properties of starch based films', *Carbohydrate Polymers*.
- Dong, S., Bortner, M. J. and Roman, M. (2016) 'Analysis of the sulfuric acid hydrolysis of wood pulp for cellulose nanocrystal production: A central composite design study', *Industrial Crops & Products*. Elsevier B.V., 93, pp. 76–87.
- Dong, X. M., Revol, J. F. and Gray, D. G. (1998) 'Effect of microcrystallite preparation conditions on the formation of colloid crystals of cellulose', *Cellulose*.
- Dufresne, A. (2012) 'Processing of nanocellulose-based materials', in Nanocellulose.
- Dungani, R., Karina, M., Subyakto, Sulaeman, A., Hermawan, D. and Hadiyane, A. (2016) 'Agricultural waste fibers towards sustainability and advanced utilization: A review', *Asian Journal of Plant Sciences*. Science Alert, 15(1–2), pp. 42–55.
- Eichhorn, S. J., Dufresne, A., Aranguren, M., Marcovich, N. E., Capadona, J. R., Rowan, S. J., Weder, C., Thielemans, W., Roman, M., Renneckar, S., Gindl, W., Veigel, S., Keckes, J., Yano, H., Abe, K., Nogi, M., Nakagaito, A. N., Mangalam, A., Simonsen, J., Benight, A. S., Bismarck, A., Berglund, L. A. and Peijs, T. (2010) 'Review: Current international research into cellulose nanofibres and nanocomposites', *Journal of Materials Science*, pp. 1–33.
- Eyley, S. and Thielemans, W. (2014) 'Surface modification of cellulose nanocrystals.', *Nanoscale*, 6(14), pp. 7764–79.
- Ferrer, A., Pal, L. and Hubbe, M. (2017) 'Nanocellulose in packaging: Advances in barrier layer technologies', *Industrial Crops and Products*. Elsevier B.V., 95, pp. 574–582.
- Flauzino Neto, W. P., Silvério, H. A., Dantas, N. O. and Pasquini, D. (2013) 'Extraction and characterization of cellulose nanocrystals from agro-industrial residue - Soy hulls', *Industrial Crops and Products*, 42(1), pp. 480–488.
- Flores, J., Kamali, M. and Ghahremaninezhad, A. (2017) 'An investigation into the properties and microstructure of cement mixtures modified with cellulose nanocrystal', *Materials*, 10(5).
- G.Venkatesan and T.Tamizhazhagan (2016) 'Ultra high strength concrete', *Science* and Technology of Concrete Admixtures, pp. 503–523.

- Gao, G., Yue, R. M., Jing, X., Liu, Z. Y. and Li, G. (2017) 'Efficient yeast cell-surface display of an endoglucanase of Aspergillus flavus and functional characterization of the whole- cell enzyme', *World Journal of Microbiology* and Biotechnology. Springer Netherlands, 33(6), pp. 1–10.
- George, J. and Sabapathi, S. N. (2015) 'Cellulose nanocrystals: Synthesis, functional properties, and applications', *Nanotechnology, Science and Applications*, 8, pp. 45–54.
- Geus, J. V. De (2014) Ultra High Performance Fibre Reinforced Concrete applied in Railway Bridges.
- Givi, A. N., Rashid, S. A., Aziz, F. N. A. and Salleh, M. A. M. (2010) 'Assessment of the effects of rice husk ash particle size on strength, water permeability and workability of binary blended concrete', *Construction and Building Materials*. Elsevier Ltd, 24(11), pp. 2145–2150.
- Goh, K. Y., Ching, Y. C., Chuah, C. H., Abdullah, L. C. and Liou, N.-S. (2016) 'Individualization of microfibrillated celluloses from oil palm empty fruit bunch : comparative studies between acid hydrolysis and ammonium persulfate oxidation', *Cellulose*. Springer Netherlands, 23(1), pp. 379–390.
- Gu, J., Hu, C., Zhong, R., Tu, D., Yun, H., Zhang, W. and Leu, S. (2017) 'Isolation of cellulose nanocrystals from medium density fiberboards', *Carbohydrate Polymers*. Elsevier Ltd.
- Hamzah, N., Tokimatsu, K. and Yoshikawa, K. (2019) 'Solid Fuel from Oil Palm Biomass Residues and Municipal Solid Waste by Hydrothermal Treatment for Electrical Power Generation in Malaysia : A Review', (3), pp. 1–23.
- Heinze, T. (2015) 'Cellulose: Structure and Properties', in Advances in Polymer Science.
- Henrique, M. A., Silvério, H. A., Flauzino Neto, W. P. and Pasquini, D. (2013) 'Valorization of an agro-industrial waste, mango seed, by the extraction and characterization of its cellulose nanocrystals', *Journal of Environmental Management*. Elsevier Ltd, 121, pp. 202–209.
- Horgnies, M., Chen, J. J. and Bouillon, C. (2013) 'Overview about the use of fourier transform infrared spectroscopy to study cementitious materials', WIT Transactions on Engineering Sciences, 77, pp. 251–262.

Hosch, W. L. (2009) 'Annual', Encyclopaedia Britannica, Inc.

- Hsu, K., Chiu, J., Chen, S. and Tseng, Y. (1999) 'Effect of addition time of a superplasticizer on cement adsorption and on concrete workability', 21, pp. 0–5.
- Hubbe, Martin A., Rojas, O. J., Lucia, L. A. and Sain, M. (2008) 'Cellulosic nanocomposites: A review', *BioResources*, 12(3), pp. 929–980.
- Hubbe, Martin A, Rojas, O. J., Lucia, L. A., Sain, M. and Forest, T. A. (2008) 'Cellulosic nanocomposites: a review', 3, pp. 929–980.
- Hughes, T. L., Methven, C. M., Jones, T. G. J., Pelham, S. E., Fletcher, P. and Hall, C. (1995) 'Determining cement composition by Fourier transform infrared spectroscopy', *Advanced Cement Based Materials*, 2(3), pp. 91–104.

International, A. (2012) AOAC: Official Methods of Analysis (Volume 1).

- ISO8301 (1991) 'Thermal Insulation Determination of Steady-State Thermal Resistance and Related PropertiesHeat Flow Meter Apparatus Available from American National Standards Institute (ANSI), 25 W. 43rd St., 4th Floor, New York, NY 10036, http://www.ansi.or', International Organization for Standardization.
- ISO9060 (2018) 'Solar energy Specification and classification of instruments for measuring hemispherical solar and direct solar radiation', *International* Organization for Standardization.
- Isogai, A. and Zhou, Y. (2019) 'Current Opinion in Solid State & Materials Science Diverse nanocelluloses prepared from TEMPO-oxidized wood cellulose fi bers: Nanonetworks, nano fi bers, and nanocrystals', *Current Opinion in Solid State & Materials Science*. Elsevier, 23(2), pp. 101–106.
- Janardhnan, S. and Sain, M. M. (2006) 'Isolation of Cellulose Microfibrils an Enzymatic Approach', *Cellulose Microfibril Isolation BioResources*.
- Jiang, B., Liu, C., Zhang, C., Wang, B. and Wang, Z. (2007) 'The effect of nonsymmetric distribution of fiber orientation and aspect ratio on elastic properties of composites', *Composites Part B: Engineering*, 38(1), pp. 24–34.
- Jiang, F., Esker, A. R. and Roman, M. (2010) 'Acid-catalyzed and solvolytic desulfation of H2SO 4-hydrolyzed cellulose nanocrystals', *Langmuir*, 26(23), pp. 17919–17925.
- Jiao, L., Su, M., Chen, L., Wang, Y., Zhu, H. and Dai, H. (2016) 'Natural cellulose nanofibers as sustainable enhancers in construction cement', *PLoS ONE*, 11(12), pp. 1–13.

- JIS A1412 (2016) 'Test method for thermal resistance and related properties of thermal insulations, Part 1: Guarded hot plate apparatus', *Japanese International Standards*.
- Johari, A., Alkali, H., Hashim, H., I. Ahmed, S. and Mat, R. (2014) 'Municipal Solid Waste Management and Potential Revenue from Recycling in Malaysia', *Modern Applied Science*, 8(4).
- Jonoobi, M., Harun, J., Mathew, A. P., Hussein, M. Z. B. and Oksman, K. (2010) 'Preparation of cellulose nanofibers with hydrophobic surface characteristics', *Cellulose*, 17(2), pp. 299–307.
- Jonoobi, M., Oladi, R. and Davoudpour, Y. (2015) 'Different preparation methods and properties of nanostructured cellulose from various natural resources and residues : a review', pp. 935–969.
- Jonoobi, M., Oladi, R., Davoudpour, Y., Oksman, K., Dufresne, A., Hamzeh, Y. and Davoodi, R. (2015) 'Different preparation methods and properties of nanostructured cellulose from various natural resources and residues: a review', *Cellulose*, 22(2), pp. 935–969.
- José, M., Bettencourt, A. and Garrido, F. (2015) 'Curing effect in the shrinkage of a lower strength self-compacting concrete', 93, pp. 1206–1215.
- Julie, J. C. S., George, N. and Narayanankutty, S. K. (2016) 'Isolation and Characterization of Cellulose Nanofibrils From Arecanut Husk Fibre', *Carbohydrate Polymers*. Elsevier Ltd.
- Junji, S., Jan, P. and Henri, C. (1991) 'Combined Infrared and Electron Diffraction Study of the Polymorphism of Native Celluloses', *Macromolecules*, 24(9), pp. 2461–2466.
- Kafle, K., Shin, H., Lee, C. M., Park, S. and Kim, S. H. (2015) 'Progressive structural changes of Avicel , bleached softwood , and bacterial cellulose during enzymatic hydrolysis', *Nature Publishing Group*. Nature Publishing Group, (May), pp. 1–10.
- Kamel, S. (2007) 'Nanotechnology and its applications in lignocellulosic composites, a mini review', *Express Polymer Letters*.
- Kargarzadeh, H., Ahmad, I., Abdullah, I., Dufresne, A., Zainudin, S. Y. and Sheltami, R. M. (2012) 'Effects of hydrolysis conditions on the morphology, crystallinity, and thermal stability of cellulose nanocrystals extracted from kenaf bast fibers', *Cellulose*, 19(3), pp. 855–866.

- Kargarzadeh, H., Ioelovich, M. and Ahmad, I. (2017) Methods for Extraction of Nanocellulose from Various.
- Kargarzadeh, H., Sheltami, R. M., Ahmad, I., Abdullah, I. and Dufresne, A. (2015) 'Cellulose nanocrystal: A promising toughening agent for unsaturated polyester nanocomposite', *Polymer (United Kingdom)*. Elsevier Ltd, 56(December 2014), pp. 346–357.
- Kavitha, B. and Thambavani, D. S. (2015) 'Characterization of Riverbed Sand from Mullai Periyar, Tamilnadu by FT-IT, XRD and SEM/EDAX', Asian Journal Of Chemistry, 27(4), pp. 1506–1508.
- Khalid, N. H. A., Hussin, M. W., Mirza, J., Ariffin, N. F., Ismail, M. A., Lee, H. S.,
  Mohamed, A. and Jaya, R. P. (2016) 'Palm oil fuel ash as potential green micro-filler in polymer concrete', *Construction and Building Materials*. Elsevier Ltd, 102, pp. 950–960.
- Khalil, H. P. S. A., Alwani, M. S. and Omar, A. K. M. (2006) 'Distribution, And Cell Wall Structure Of Malaysian Plant Waste Fibers', 1, pp. 220–232.
- Khushefati, W. H. and Demirbog, R. (2015) 'Effects of nano and micro size of CaO and MgO, nano-clay and expanded perlite aggregate on the autogenous shrinkage of mortar', 81, pp. 268–275.
- Kimura, S. and Itoh, T. (1996) 'New cellulose synthesizing complexes (terminal complexes) involved in animal cellulose biosynthesis in the tunicate Metandrocarpa uedai', *Protoplasma*.
- Kochova, K., Schollbach, K. and Brouwers, H. J. (2016) 'Use of alternative organic fibres in cement composites.', 4th International Conference on Sustainable Solid Waste Management, (June), pp. 1–8.
- Kruyeniski, J., Ferreira, P. J. T., Videira, G. and Carvalho, S. (2019) 'Industrial Crops & Products Physical and chemical characteristics of pretreated slash pine sawdust influence its enzymatic hydrolysis', *Industrial Crops & Products*. Elsevier, 130(July 2018), pp. 528–536.
- Kumar, A., Negi, Y. S., Choudhary, V. and Bhardwaj, N. K. (2014) 'Characterization of Cellulose Nanocrystals Produced by Acid-Hydrolysis from Sugarcane Bagasse as Agro-Waste', *Journal of Materials Physics and Chemistry*, 2(1), pp. 1–8.

- Kvien, I., Tanem, B. S. and Oksman, K. (2005) 'Characterization of cellulose whiskers and their nanocomposites by atomic force and electron microscopy', *Biomacromolecules*, 6(6), pp. 3160–3165.
- Lam, E., Male, K. B., Chong, J. H., Leung, A. C. W. and Luong, J. H. T. (2012) 'Applications of functionalized and nanoparticle-modified nanocrystalline cellulose', *Trends in Biotechnology*. Elsevier Ltd, 30(5), pp. 283–290.
- Leng, T. (2016) 'Cellulose Nanocrystals : Particle Size Distribution and Dispersion in Polymer Composites Tianyang Leng MSc degree in Chemistry'.
- Leszczyńska, A., Radzik, P., Haraźna, K. and Pielichowski, K. (2018) 'Thermal stability of cellulose nanocrystals prepared by succinic anhydride assisted hydrolysis', *Thermochimica Acta*. Elsevier B.V., 663, pp. 145–156.
- Li, C., Li, Q., Ni, X., Liu, G., Cheng, W. and Han, G. (2017) 'Coaxial electrospinning and characterization of core-shell structured cellulose nanocrystal reinforced PMMA/PAN composite fibers', *Materials*, 10(6).
- Li, R., Fei, J., Cai, Y., Li, Y., Feng, J. and Yao, J. (2009) 'Cellulose whiskers extracted from mulberry: A novel biomass production', *Carbohydrate Polymers*. Elsevier Ltd, 76(1), pp. 94–99.
- Lin, K., Enomae, T. and Chang, F. (2019) 'Cellulose Nanocrystal Isolation from Hardwood Pulp', *molecules*, 24(3724), pp. 3–5.
- Ling, Y.-F., Zhang, P., Wang, J. and Shi, Y. (2020) 'Effect of Sand Size on Mechanical Performance of Cement-Based Composite Containing PVA Fibers and Nano-SiO2', *Materials*, pp. 1–14.
- Lu, P. and Hsieh, Y.-L. (2010) 'Preparation and properties of cellulose nanocrystals: Rods, spheres, and network', *Carbohydrate Polymers*. Elsevier Ltd., 82(2), pp. 329–336.
- Lu, P. and Lo, H. Y. (2010) 'Preparation and properties of cellulose nanocrystals: Rods, spheres, and network', *Carbohydrate Polymers*.
- Madsen, B. and Gamstedt, E. K. (2013) 'Wood versus plant fibers: Similarities and differences in composite applications', *Advances in Materials Science and Engineering*, 2013.
- Malaysian Palm Oil Board (2019) Monthly Production Of Oil Palm Products Summary For The Month Of September 2019.
- Mao, J., Abushammala, H., Brown, N. and Laborie, M. (2017) 'Comparative Assessment of Methods for Producing Cellulose I Nanocrystals from

Cellulosic Sources', in *Nanocelluloses: Their Preparation, Properties, and Applications*, pp. 19–53.

- Marchessault, R. H., Morehead, F. F. and Walter, N. M. (1959) 'Liquid crystal systems from fibrillar polysaccharides', *Nature*.
- Mariano, M., El Kissi, N. and Dufresne, A. (2014) 'Cellulose nanocrystals and related nanocomposites: Review of some properties and challenges', *Journal of Polymer Science Part B: Polymer Physics*, 52(12), pp. 791–806.
- Martínez-Barrera, G., Gencel, O., Reis, J. M. L. D. and Del Coz Diáz, J. J. (2014) 'Novel Technologies and Applications for Construction Materials', *Advances in Materials Science and Engineering*, 2014, pp. 2–4.
- Mascheroni, E., Rampazzo, R., Aldo, M., Piva, G., Bonetti, S. and Piergiovanni, L. (2016) 'Comparison of cellulose nanocrystals obtained by sulfuric acid hydrolysis and ammonium persulfate, to be used as coating on flexible foodpackaging materials', *Cellulose*. Springer Netherlands, 23(1), pp. 779–793.
- Mathew, A. P. and Dufresne, A. (2002) 'Morphological investigation of nanocomposites from sorbitol plasticized starch and tunicin whiskers', *Biomacromolecules*, 3(3), pp. 609–617.
- Mazlan, D. and Awal, A. S. M. A. (2012) 'Properties of Cement Based Composites Containing Oil Palm Stem as Fiber Reinforcement', *Malaysian Journal of Civil Engineering*, 24(2), pp. 107–117.
- McDonald, C., Glasser, F. P. and Imbabi, M. S. (2019) 'A New, Carbon-Negative Precipitated Calcium Carbonate Admixture (PCC-A) for Low Carbon Portland Cements', *Materials*, 12.
- Mehra, P., Gupta, R. C. and Thomas, B. S. (2016) 'Properties of concrete containing jarosite as a partial substitute for fine aggregate', *Journal of Cleaner Production*. Elsevier Ltd, 120, pp. 241–248.
- Menéndez, G., Bonavetti, V. and Irassar, E. F. (2003) 'Strength development of ternary blended cement with limestone filler and blast-furnace slag', *Cement and Concrete Composites*, 25(1), pp. 61–67.
- Min, F., Yao, Z. and Jiang, T. (2014) 'Experimental and numerical study on tensile strength of concrete under different strain rates.', *TheScientificWorldJournal*, 2014, p. 173531.

- Mohammed, L., Ansari, M. N. M., Pua, G., Jawaid, M. and Islam, M. S. (2015) 'A Review on Natural Fiber Reinforced Polymer Composite and Its Applications', 2015.
- Moon, R. J., Martini, A., Nairn, J., Simonsen, J. and Youngblood, J. (2011) 'Cellulose nanomaterials review: structure, properties and nanocomposites.', *Chemical Society reviews*, 40(7), pp. 3941–3994.
- Negar, K. and Milad, F. (2017) 'Production of cellulose nanocrystals from pistachio shells and their application for stabilizing Pickering emulsions', *International Journal of Biological Macromolecules*. Elsevier B.V.

Neville, A. M. (2006) Concrete Technology. Pearson Education.

- Nishiyama, Y., Sugiyama, J., Chanzy, H. and Langan, P. (2003) 'Crystal Structure and Hydrogen Bonding System in Cellulose Iα from Synchrotron X-ray and Neutron Fiber Diffraction', *Journal of the American Chemical Society*. American Chemical Society, 125(47), pp. 14300–14306.
- Njoku, R. E. and Obikwelu, D. O. N. (2008) 'Swelling Characteristics and Tensile Properties of Natural Fiber Reinforced Plastic in Selected Solvents', *Nigerian Journal of Technology*, 27(2), pp. 58–63.
- O'Sullivan, A. C. (1997) 'Cellulose: the structure slowly unravels', *Cellulose*, 4(3), pp. 173–207.
- Oke, I. (2010) 'Nanoscience in nature: cellulose nanocrystals', 3(2), pp. 77-80.
- Opara, H. E., Eziefula, U. G. and Eziefula, B. I. (2018) 'Comparison of physical and mechanical properties of river sand concrete with quarry dust concrete', *Selected Scientific Papers Journal of Civil Engineering*, 13(s1), pp. 127–134.
- Pandey, J. K., Kim, C. S., Chu, W. S., Lee, C. S., Jang, D. Y. and Ahn, S. H. (2009) 'Evaluation of morphological architecture of cellulose chains in grass during conversion from macro to nano dimensions', *E-Polymers*.
- Pane, I. and Hansen, W. (2005) 'Investigation of blended cement hydration by isothermal calorimetry and thermal analysis', *Cement and Concrete Research*, 35(6), pp. 1155–1164.
- Parveen, S., Rana, S. and Fangueiro, R. (2013) 'A review on nanomaterial dispersion, microstructure, and mechanical properties of carbon nanotube and nanofiber reinforced cementitious composites', *Journal of Nanomaterials*, 2013.

- Pelissari, F. M., Sobral, P. J. D. A. and Menegalli, F. C. (2014) 'Isolation and characterization of cellulose nanofibers from banana peels', *Cellulose*, 21(1), pp. 417–432.
- Peng, B. L., Dhar, N., Liu, H. L. and Tam, K. C. (2011) 'Chemistry and applications of nanocrystalline cellulose and its derivatives: A nanotechnology perspective', *The Canadian Journal of Chemical Engineering*, 89(5), pp. 1191–1206.
- Pereda, M. and Dufresne, A. (2014) 'Cellulose nanocrystals and related polymer nanocomposites', *Biomass-based Biocomposites*, (January 2013), pp. 305– 348.
- Peters, S. J., Rushing, T. S., Landis, E. N. and Cummins, T. K. (2010) 'Nanocellulose and Microcellulose Fibers for Concrete', *Transportation Research Record: Journal of the Transportation Research Board*, 2142(1), pp. 25–28.
- Petter Jelle, B. (2016) Nano-based thermal insulation for energy-efficient buildings, Start-Up Creation: The Smart Eco-Efficient Built Environment. Elsevier Ltd.
- Pickering, K. L., Efendy, M. G. A. and Le, T. M. (2016) 'A review of recent developments in natural fibre composites and their mechanical performance', *Composites Part A: Applied Science and Manufacturing*, 83, pp. 98–112.
- Plasser, W. and Rahbaran, S. (2017) 'Biodegradability of wood-based cellulose fibers The Lenzing Group 2016'.
- Poletto, M., Júnior, H. L. O. and Zattera, A. J. (2014) 'Native Cellulose: Structure, Characterization and Thermal Properties', *Materials*, 7(May 2015), pp. 6105– 6119.
- Poole, C. P. and Owens, F. J. (2013) Introduction To, Introduction to Nanotechnology.
- Rafi, A. S. M. M., Tasnim, U. F. and Rahman, M. S. (2018) 'Quantification and Qualification of Silica Sand Extracted from Padma River Sand', *IOP Conference Series: Materials Science and Engineering*, 438(1).
- Rahuman, A. and Yeshika, S. (2015) 'Study On Properties Of Sisal Fiber Reinforced Concrete With Different Mix Proportions And Different Percentage Of Fiber Addition', pp. 2319–2322.
- Ramezani, M. G. and Golchinfar, B. (2019) 'Mechanical Properties of Cellulose Nanocrystal (CNC) Bundles: Coarse-Grained Molecular Dynamic Simulation', *Journal of Composites Science*, 3(2), p. 57.

- Ranby, B. G. (1951) 'The Colloidal Properties Of Cellulose Micelles', Fibrous Macromolecular Systems, Iv(111), pp. 158–164.
- Rantuch, P. and Chrebet, T. (2014) 'Thermal decomposition of cellulose insulation', *Cellulose Chemistry and Technology*, 48(5–6), pp. 461–467.
- Rathore, A. and Pradhan, M. K. (2017) 'Hybrid Cellulose Bionanocomposites from banana and jute fibre: A Review of ScienceDirect Hybrid Cellulose Bionanocomposites from banana and jute fibre: A Review of Preparation, Properties and Applications', *Materials Today: Proceedings*. Elsevier Ltd, 4(2), pp. 3942–3951.
- Reiniati, I. (2017) Bacterial Cellulose Nanocrystals: Production and Application.
- Rosa, M. F., Medeiros, E. S., Malmonge, J. a., Gregorski, K. S., Wood, D. F., Mattoso,
  L. H. C., Glenn, G., Orts, W. J. and Imam, S. H. (2010) 'Cellulose nanowhiskers from coconut husk fibers: Effect of preparation conditions on their thermal and morphological behavior', *Carbohydrate Polymers*. Elsevier Ltd, 81(1), pp. 83–92.
- Sajjala, K. (2018a) 'A review on natural fibres in the concrete', (September).
- Sajjala, K. (2018b) 'A review on natural fibres in the concrete', (April 2017).
- Satyamurthy, P., Jain, P., Balasubramanya, R. H. and Vigneshwaran, N. (2011)
  'Preparation and characterization of cellulose nanowhiskers from cotton fibres by controlled microbial hydrolysis', *Carbohydrate Polymers*. Elsevier Ltd., 83(1), pp. 122–129.
- Saxena, A. (2013) Nanocomposites Based On Nanocellulose Whiskers.
- Saxena, I. M. and Brown, R. M. (2005) 'Cellulose biosynthesis: Current views and evolving concepts', *Annals of Botany*, 96(1), pp. 9–21.
- Science, M. and Western, C. (1976) 'Determination of the Structure of Cellulose II', *Macromolecules*, 9(2).
- Shahedan, N. F., Mustafa, M. and Bakri, A. (2017) 'Review on thermal insulation performance in various type of concrete Review on Thermal Insulation Performance in Various Type of Concrete', 020046(April).
- Sheltami, R., Kargarzadeh, H., Abdullah, I. and Ahmad, I. (2017) 'Thermal Properties of Cellulose Nanocomposites', *Journal of Thermal Analysis*, 35(7), pp. 2235– 2242.
- Shen, D., Jiang, J., Shen, J., Yao, P. and Jiang, G. (2016) 'Influence of curing temperature on autogenous shrinkage and cracking resistance of high-

performance concrete at an early age', *Construction and Building Materials*. Elsevier Ltd, 103, pp. 67–76.

- Silvério, H. A., Flauzino Neto, W. P., Dantas, N. O. and Pasquini, D. (2013) 'Extraction and characterization of cellulose nanocrystals from corncob for application as reinforcing agent in nanocomposites', *Industrial Crops and Products*. Elsevier B.V., 44, pp. 427–436.
- Siqueira, G., Bras, J. and Dufresne, A. (2010a) 'Cellulosic Bionanocomposites: A Review of Preparation, Properties and Applications', *Polymers*, 2(4), pp. 728– 765.
- Siqueira, G., Bras, J. and Dufresne, A. (2010b) 'New process of chemical grafting of cellulose nanoparticles with a long chain isocyanate', *Langmuir*.
- Soltan, D. G., Olvera, A., Savastano, H. and Li, V. C. (2017) 'Introducing a curauá fiber reinforced cement-based composite with strain-hardening behavior Tensile Stres s 3 (MPa) Polyvinylalcohol (PVA) Polyeth ylene (PE) Polypropylene (HTPP) Strain (%)', *Industrial Crops & Products*. Elsevier B.V., 103, pp. 1–12.
- Song, X., Zhou, L., Ding, B., Cui, X., Duan, Y. and Zhang, J. (2018) 'Simultaneous improvement of thermal stability and redispersibility of cellulose nanocrystals by using ionic liquids', *Carbohydrate Polymers*. Elsevier, 186(January), pp. 252–259.
- De Souza Lima, M. M. and Borsali, R. (2004) 'Rodlike cellulose microcrystals: Structure, properties, and applications', *Macromolecular Rapid Communications*.
- Springfield and Tyler (2011) Application of FTIR for Quantification of Alkali in Cement, Master of Science (Engineering Systems – Mechanical Systems).
- Stutzman, P. (2004) 'Scanning electron microscopy imaging of hydraulic cement microstructure', *Cement and Concrete Composites*.
- Sudin, R. and Swamy, N. (2006) 'Bamboo and wood fibre cement composites for sustainable infrastructure regeneration', *Journal of Materials Science*, 41(21), pp. 6917–6924.
- Sugiyama, J., Vuong, R. and Chanzy, H. (1991) 'Electron-Diffraction Study on the 2 Crystalline Phases Occurring in Native Cellulose From an Algal Cell-Wall', *Macromolecules*, 24(14), pp. 4168–4175.

- Suksawang, N., Wtaife, S. and Alsabbagh, A. (2018) 'Evaluation of Elastic Modulus of Fiber-Reinforced Concrete', *Aci Materials Journal*, (March), pp. 239–249.
- Sun, B., Zhang, M., Hou, Q., Liu, R., Wu, T. and Si, C. (2016) 'Further characterization of cellulose nanocrystal (CNC) preparation from sulfuric acid hydrolysis of cotton fibers', *Cellulose*. Springer Netherlands, 23(1), pp. 439– 450.
- Swann, G. E. A. and Patwardhan, S. V. (2011) 'Application of Fourier Transform Infrared Spectroscopy (FTIR) for assessing biogenic silica sample purity in geochemical analyses and palaeoenvironmental research', *Climate of the Past*, 7(1), pp. 65–74.
- Taheri, A. and Mohammadi, M. (2015) 'The use of cellulose nanocrystals for potential application in topical delivery of hydroquinone', *Chemical Biology and Drug Design*, 86(1), pp. 882–886.
- Tamai, H. (2015) 'Enhancing the performance of porous concrete by utilizing the pumice aggregate', *Procedia Engineering*. Elsevier B.V., 125, pp. 732–738.
- Tayeb, A. H., Amini, E., Ghasemi, S. and Tajvidi, M. (2018) 'Cellulose Nanomaterials
   Binding Properties and Applications : A Review', *molecules*, 23(2684), pp. 1–24.
- Tian, C., Yi, J., Wu, Y., Wu, Q., Qing, Y. and Wang, L. (2016) 'Preparation of highly charged cellulose nanofibrils using high-pressure homogenization coupled with strong acid hydrolysis pretreatments', *Carbohydrate Polymers*. Elsevier Ltd., 136, pp. 485–492.
- Tian, L., Chen, J. and Yu, S. (2013) 'How has Shenzhen been heated up during the rapid urban build-up process?', *Landscape and Urban Planning*. Elsevier B.V., 115, pp. 18–29.
- Tosic, N., Marinkovic, S. and Stojanovic, A. (2017) 'Sustainability of the concrete industry: Current trends and future outlook', *Tehnika*, 72(1), pp. 38–44.
- Trache, D., Hussin, M. H., Haafiz, M. K. M. and Thakur, V. K. (2017) 'Recent progress in cellulose nanocrystals: sources and production', *Nanoscale*.
- Wang, G., Yu, D., Kelkar, A. D. and Zhang, L. (2017) 'Electrospun nanofiber: Emerging reinforcing filler in polymer matrix composite materials', *Progress* in Polymer Science. Elsevier Ltd, 75, pp. 73–107.

- Wang, J., Van Tittelboom, K., De Belie, N. and Verstraete, W. (2012) 'Use of silica gel or polyurethane immobilized bacteria for self-healing concrete', *Construction and Building Materials*. Elsevier Ltd, 26(1), pp. 532–540.
- Wang, S. and Li, V. C. (2006) 'High-early-strength engineered cementitious composites', 103(2), p. 9.
- Wang, Y., Cao, X. and Zhang, L. (2006) 'Effects of cellulose whiskers on properties of soy protein thermoplastics', *Macromolecular Bioscience*, 6(7), pp. 524–531.
- Xu, Y., Kraemer, D., Song, B., Jiang, Z., Zhou, J., Loomis, J., Wang, J., Li, M., Ghasemi, H., Huang, X., Li, X. and Chen, G. (2019) 'Nanostructured polymer films with metal-like thermal conductivity', *Nature Communications*. Springer US, 10(1771), pp. 1–8.
- Yamane, C. and Hirase, R. (2015) 'Mechanism of structure formation and dissolution of regenerated cellulose from cellulose / aqueous sodium hydroxide solution and formation of molecular sheets deduced from the mechanism', *Cellulose*. Springer Netherlands, 22(5), pp. 2971–2982.
- Yang, S., Chen, G., Lv, C., Li, C., Yin, N., Yang, F. and Xue, L. (2018) 'Evolution of nanopore structure in lacustrine organic-rich shales during thermal maturation from hydrous pyrolysis, Minhe Basin, Northwest China', *Energy Exploration* and Exploitation, 36(2), pp. 265–281.
- Yu, H., Qin, Z., Liang, B., Liu, N., Zhou, Z. and Chen, L. (2013) 'Facile extraction of thermally stable cellulose nanocrystals with a high yield of 93% through hydrochloric acid hydrolysis under hydrothermal conditions', *Journal of Materials Chemistry A*, 1(12), p. 3938.
- Yu, H. Y., Zhang, D. Z., Lu, F. F. and Yao, J. (2016) 'New Approach for Single-Step Extraction of Carboxylated Cellulose Nanocrystals for Their Use As Adsorbents and Flocculants', ACS Sustainable Chemistry and Engineering, 4(5), pp. 2632–2643.
- Zeyad, A. M., Megat Johari, M. A., Tayeh, B. A. and Yusuf, M. O. (2017) 'Pozzolanic reactivity of ultrafine palm oil fuel ash waste on strength and durability performances of high strength concrete', *Journal of Cleaner Production*. Elsevier Ltd, 144, pp. 511–522.
- Zhang, P., Li, Q., Chen, Y., Shi, Y. and Ling, Y.-F. (2019) 'Durability of Steel Fiber-Reinforced Concrete', *Materials*, 12.

- Zhao, J., Lu, C., He, X., Zhang, Xiaofang, Zhang, W. and Zhang, Ximu (2015) 'Polyethylenimine-grafted cellulose nanofibril aerogels as versatile vehicles for drug delivery', ACS Applied Materials and Interfaces, 7(4), pp. 2607–2615.
- Zhou, C. and Wu, Q. (2012) 'Recent Development in Applications of Cellulose Nanocrystals for Advanced Polymer-Based Nanocomposites by Novel Fabrication Strategies'.
- Zhu, H., Shen, F., Luo, W., Zhb, S., Zhao, M., Natarajan, B., Dai, J., Ji, X., Yassar, R.
   S. and Li, T. (2017) 'Low Temperature Carbonization of Cellulose Nanocrystals for High Performance Carbon Anode of Sodium-Ion Batteries', *Nano Energy*. Elsevier.

## LIST OF PUBLICATION

## JOURNAL PUBLICATION

- Mazlan, D., Krishnan, S., Din, M.F.M. et al. Effect of Cellulose Nanocrystals Extracted from Oil Palm Empty Fruit Bunch as Green Admixture for Mortar. Sci Rep 10, 6412, 2020 (Q1 Journal, Impact Factor 5.0)
- D. Mazlan, M. F. Md Din, C. Tokoro, and I. S. Ibrahim, "Cellulose Nanocrystals Addition Effects on Cement Mortar Matrix Properties," Int. J. Adv. Mech. Civ. Eng., vol. 3, no. 1, pp. 44–48, 2016 (Scopus Indexed)

## **CONFERENCE/ PROCEEDING PUBLICATION**

- Zanariah Jahya, Zaiton Haron, Musli Nizam Yahya, Dianah Mazlan and Nadirah Darus. "Preliminary investigation of acoustical proeperties of concrete containing oil palm shellas an aggregate replacement," *International conference on Civil & EnvironmentalEngineering 2017 (CENVIRON 2017),* Penang, Malaysia, 28-29 November 2017.
- D. Mazlan, M.F. Md Din, C. Tokoro, I.S. Ibrahim. "Cellulose Nanocrystals Addition Effects On Cement Mortar Matrix Properties" *International Conference on Civil and Environmental Engineering (I2C2E)*, pp. 1-5. Osaka, Japan, December 9th, 2015.
- 3. Ooi Theam Yiew, Dianah Mazlan, Mohd Fadhil Md Din, Azlan Abdul Rahman,Nur Suraya Kamsano., "Review On Urban Heat Island Mitigation Techniques And Impacts", International Conference on Sustainability Initiatives (ICSI) 2015 in conjunction with 8<sup>th</sup> ASEAN Environmental Engineering Conference (AEEC), Kuala Lumpur, Malaysia, 24- 25 August 2015