# DEVELOPMENT OF SUSTAINABLE CONCRETE USING IRON ORE TAILINGS AS SAND REPLACEMENT

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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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> > JANUARY 2017

### **DEDICATION**

I dedicated this work to:

My mother, **Hajja Kaltum Shettima** whose sacrifice; My Late Uncle, **Alhaji Goni Muhammad Bama**, whose dream; My **Father, Brothers and sisters**, whose support and encouragement; And My beloved wife, **Amina Lawan Abdu**, whose love and patience;

led to achieving my doctoral degree

#### ACKNOWLEDGEMENT

First and foremost all Praise be To Allah (S.W.T) for the benefit of wisdom and power to make this work possible and overcome this long academic pursuit.

I would like to express my sincere appreciation and gratitude to my supervisor Prof. Ir. Dr. Mohd Warid Bin Hussin for his support, guidance, encouragement and patience throughout this research period. Without his unwavering guidance, support, and valuable advice during the research and writing, this thesis would have been incomplete. Furthermore, I would like to extend my gratitude to my co-supervisor, Assoc. Prof. Dr. Yusof Ahmad for his generous time, fruitful discussions, motivation, and patience to attend to my numerous questions during this study.

My appreciation goes to the technical staff of Structures and concrete materials laboratory for attending to my various laboratory works. I am very grateful to my research colleagues, especially Dr. Ibrahim Abubakar Shehu, Dr. Ibrahim Ogiri Hassan, Dr. Habib Alh. Bukar, Dr. Mostafa Samadi, Dr. Nur Faharyu Binti Ariffin, Dr. Nor Hasanah Abdul Shukor Lim, Dr. Nur Hafizah Binti Abd Khalid and many others. Same goes to my friends and brothers such as Usman Goni Bama, Umar Ali Alkali, Ali Dauda Abubarkar, Ma,aji Abba Bashir, Dr. Jafaru Usman, Dr. Kallamu Usman, Mal. Umar Barde, Madaki Mamu Abdullahi, Abba Mustafa, and many others.

I remain immensely grateful for the financial support by the management of Federal Polytechnic, Damaturu, Nigeria, through Federal Government of Nigeria Education Trust Fund.

Finally, my special thanks to my beloved parents, brothers and sisters for their prayers unending love, sacrifice, encouragement and support. The same goes to my children and wife Amina Lawan Abdu for their unreserved support, love, and patience towards the success of this thesis.

#### ABSTRACT

The increasing demands for iron ore worldwide have resulted in the generation of billion tonnes of iron ore tailings (IOT) which were found in all the iron ore mining industries. Rapid increase in consumption of river sand due to the increased in construction activities over exploited the riverbeds. This has led to a range of problems which include: depletion of natural sand, increased riverbed depth, water table lowering, intrusion of salinity and destruction of river embankment. This study explored the possibility of using IOT as a replacement for natural river sand in concrete production. Laboratory investigations were conducted to evaluate the characterization of IOT materials in terms of microstructure, physical and chemical properties. Leaching behaviour of IOT materials was also determined. Furthermore, mix design and the evaluation of the fresh and hardened properties of the IOT concrete were executed. Series of concrete were prepared with IOT at a replacement level of 25%, 50%, 75% and 100%, using water to cement ratio (w/c) of 0.40 and 0.60. Fresh properties of mixtures in terms of concrete slumps and density were studied. The hardened properties examined are mechanical strengths, deformation characteristics, durability properties and corrosion measurement. Corrosion rate were evaluated using linear polarization techniques. Finally, microstructural tests in terms of X-ray Diffraction (XRD), Field emission scanning microscopy (FESEM), Fourier Transform Infrared Spectroscopy (FTIR) and Thermo gravimetric analysis (TGA) were concurrently conducted on control and IOT concrete in order to determine the interaction and effect of the IOT material that brings about the performance of the concrete. A correlation coefficient using fitted linear regression analysis was performed on compressive strength to evaluate the significant level of concretes containing IOT. Results showed that IOT affect mixture workability negatively. However, the inclusion of super plasticiser showed tremendous influence in increasing the workability and reduced this drawback. At 50% replacement, the compressive strength of the concrete at 28 days was 65.6 and 37.7 MPa for 0.40 and 0.60 w/c ratio, respectively, which shows an improvement of 9% and 12% over the concrete with river sand. Concrete with IOT indicates a good resistance to carbonation compared to control specimen. Linear polarization resistance (LPR) results indicates that, corrosion rates of 0.02 mm/year for IOT concretes were the same with control at 0.60 w/c ratio while 0.01 mm/year was observed for control at 0.40 w/c ratio. Considering all these test results, 50% river sand replacement with IOT resulted in concrete of excellent strength and adequate durability performance except for exposure to acid attack. However, it has the quality to be used as partial replacement of sand.

#### ABSTRAK

Permintaan yang semakin meningkat untuk bijih besi di seluruh dunia telah menyebabkan terhasilnya berbilion tan tahi bijih besi (IOT) yang ditemui dalam industri perlombongan bijih besi. Peningkatan pesat dalam penggunaan pasir sungai adalah disebabkan oleh peningkatan dalam aktiviti pembinaan. Ini telah membawa kepada pelbagai masalah termasuk pengurangan pasir, peningkatan kedalaman dasar sungai, penurunan aras air bumi, perubahan kemasinan dan pemusnahan tambak sungai. Kajian ini meneroka kemungkinan menggunakan IOT sebagai pengganti pasir sungai semula jadi dalam pengeluaran konkrit. Penyelidikan makmal telah dijalankan untuk menilai pencirian bahan IOT dari segi sifat mikrostruktur, fizikal dan kimia. Tingkah laku peresapan bahan IOT ditentukan. Tambahan pula, kaedah pendekatan reka bentuk campuran dan penilaian sifat-sifat segar dan keras konkrit IOT juga dilaksanakan. Beberapa siri sampel konkrit IOT telah disediakan untuk pelbagai peringkat penggantian pasir iaitu 25%, 50%, 75% dan 100%, menggunakan nisbah airsimen (w/c) 0.40 dan 0.60. Sifat segar campuran seperti kejatuhan dan ketumpatan telah dikaji. Sifat konkrit keras juga diperiksa seperti kekuatan mekanikal, ciri-ciri ubah bentuk, ciri-ciri ketahanan dan pengukuran kakisan. Kadar kakisan telah dinilai menggunakan teknik polarisasi linear. Akhir sekali, ujian mikrostruktur dari segi Xray Diffraction (XRD), pengimbasan pelepasan mikroskop (FESEM), jelmaan Fourier spektroskopi inframerah (FTIR) dan analisis termogravimetri (TGA) telah dijalankan ke atas konkrit kawalan dan konkrit IOT untuk menentukan interaksi dan kesan bahan IOT yang menentukan prestasi konkrit. Pekali korelasi menggunakan analisis regresi linear telah dilakukan ke atas kekuatan mampatan untuk menilai tahap ketara konkrit yang mengandungi IOT. Hasil kajian menunjukkan bahawa IOT memberi kesan negatif terhadap kebolehkerjaan. Walau bagaimanapun, penambahan bahun superplastik telah menunjukkan pengaruh yang besar dalam meningkatkan kebolehkerjaan dan mengurangkan kelemahan ini. Untuk penggantian 50%, kekuatan mampatan konkrit pada 28 hari adalah 65.6 dan 37.7 MPa masing-masing untuk nisbah w/c 0.40 dan 0.60, yang menunjukkan peningkatan sebanyak 9% dan 12% berbanding konkrit dengan pasir sungai. Konkrit dengan IOT menunjukkan rintangan yang baik untuk pengkarbonan berbanding spesimen kawalan. Keputusan rintangan polarisasi Linear (LPR) menunjukkan bahawa, kadar kakisan 0.02 mm / tahun untuk konkrit IOT adalah sama dengan kawalan pada nisbah w/c 0.60 manakala 0.01 mm/tahun untuk kawalan pada nisbah w/c 0.40. Mengambil kira semua keputusan ujian, 50% penggantian pasir sungai dengan IOT menyebabkan konkrit mempunyai kekuatan yang mencukupi dengan ketahanlasakan yang baik kecuali jika terdedah kepada serongan asid. Walau bagaimanapun, bahan ini sesuai untuk digunakan sebangai bahan separa pengganti pasir.

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## LIST OF ABBREVIATIONS

ACI	-	American Concrete Institute
ASTM	-	American Society for Testing and Materials
BS	-	British Standard
C-A-S-H	-	Calcium alumina silicate hydrate
C-S-H	-	Calcium silicate hydrate
Ca	-	Calcium
CaCO <sub>3</sub>	-	Calcium carbonate
CaO	-	Calcium oxide
Ca(OH) <sub>2</sub>	-	Calcium hydroxide
$CO_2$	-	Carbon dioxide
DTA	-	Differential Thermal Analysis
Ε	-	Ettringite
EDX	-	Energy Dispersive Electron Microscope
FESEM	-	Field Emission Scanning Electron Microscope
FTIR	-	Fourier Transform Infrared Spectroscopy
$H_2SO_4$	-	Sulphuric Acid
ICP-MS	-	Inductive Couple Plasma Mass Spectrometry
ΙΟΤ	-	Iron ore tailings
KBR	-	Potassium Bromide
LOI	-	Loss of ignition
LPR	-	Linear polarization resistance
LVDT	-	Linear Variable Differential Transducer
MOE	-	Modulus of Elasticity
NaCl	-	Sodium Chloride
NaOH	-	Sodium Hydroxide
OPC	-	Ordinary Portland cement

Q	-	Quartz
SCE	-	Saturated Calomel Electrode
SiO <sub>2</sub>	-	Silica
SP	-	Super plasticizer
TGA	-	Thermo Gravimetric Analysis
UPV	-	Ultrasonic Pulse Velocity
US EPA	-	United State Environmental Protection Agency
XRD	-	X-Ray Diffraction
XRF	-	X-Ray Fluorescence Spectrometer

## LIST OF SYMBOLS

А	-	Cross sectional area
D	-	Specimen density
f <sub>30</sub>	-	Compressive strength of grade 30 concrete
f <sub>60</sub>	-	Compressive strength of grade 60 concrete
f <sub>ct</sub>	-	Tensile strength
$\mathbf{f}_{cf}$	-	Flexural strength
$f_{cu}$	-	Compressive strength
Fe	-	Iron
Io	-	current
i <sub>corr</sub>	-	Corrosion current density
Icorr	-	Total anodic current
Ø	-	Diameter
μ	-	Micron
$m_1$	-	Mass of container
$m_2$	-	Mass of container with fresh concrete
Q	-	Total charge passed
W	-	Percentage of water absorption
$\mathbf{W}_{d}$	-	Weight of specimen dry
$W_{w}$	-	Weight of specimen wet
V	-	Volume of container

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### **CHAPTER 1**

#### **INTRODUCTION**

### 1.1 General appraisal

Malaysia is currently moving from the developing country towards achieving a developed nation status as envisaged by the year 2020. Many of the activities and industrial development are of considerable significance to the realization of this vision. Meanwhile, the socio-economic development is being actively planned and carried out. Thus, the construction sector is considered to be one of the most vital industries to sustain the overall economic growth of Malaysia by providing the basic physical infrastructure needed to support the economic development activities. The industry also plays a significant role in creating housing for the annually growing Malaysian population. According to plan, the construction sector is predicted to boom and grow rapidly in the coming years, considering the present construction scenario in Malaysia. The rapid growth of this sector, in conjunction with economic growth, consequently indirectly requires considerably high amount of production and consumption of construction minerals, such as rock materials (aggregate) and sand.

However, the environmental concern is currently rising as one of the main challenging issues affecting the natural concrete aggregate production. The consumption of natural aggregate concrete as the largest component material is a key concern because it comprises 70 to 80% of the total volume (Neville, 2011). More than 10 billion tonnes of concrete were produced annually worldwide (Yaprak et al., 2011). The high demand of aggregate for the production of concrete requires massive use of natural aggregate which will destroy the ecological balance of the environment. These include depletion of virgin aggregate deposits.

Sand is generally used as fine aggregate in concrete and usually produced from mining exploration. Sand mining is of great importance to the Malaysian economy. It should however, be recognised that the processes of prospecting, extracting and transporting have great potential for disrupting the natural environment (Rabie et al., 1994). Physical impacts of sand mining include reduction of water quality and destabilization of the stream bed and banks. Sand mining can also disrupts sediment supply and channel form, which can result in a deepening of the channel as well as sedimentation of habitats downstream. Channel instability and sedimentation from instream mining also can damage public infrastructure (bridges, pipelines, and utility lines). This process can also destroy riverine vegetation, cause erosion, pollute water sources and reduce the diversity of animals supported by these woodlands habitats (Byrnes and Hiland, 1995). In view of that, there is urgent need to find alternative replacements for river sand as fine aggregate in concrete by exploring the use of industrial waste in the making of concrete.

The challenge for the civil engineering community with the concept of sustainable development involves the use of waste materials and by-products at reasonable cost with the lowest possible environmental impact. The entrenchment of sustainable environment has become very important in order to preserve valued environmental conditions as well as available resources. As part of measures toward fostering sustainability; the reduction in emissions of greenhouse gasses, the depletion of fossil fuel and waste generation and disposal has become critical issues for consideration. The construction industry through its activities has strong impact on the environment. Although these impacts are both positive and negative, the latter gives an underlying motive for the concern to seek mitigations for environmental problems.

Currently, achieving a sustainable environment and eco-friendly community through effective recycling of waste materials in the construction industries are the fundamental issues worldwide. The utilization of certain categories of waste and byproducts of industries in construction and as building materials for the production of concrete seems to provide adequate solutions to these issues. Various researches have broadly proven that waste materials from industries such as foundry sand, copper tailings, recycled concrete, fly ash, and ceramic waste can be utilized in the production of sustainable concretes. However, the increasing amount of industrial wastes being produced due to the rapid increase in industrialization worldwide has dwindled the space for landfill. This problem of landfill and other economic and environmental issues clamour for more usage of industrial waste materials through extensive research and utilization in concrete to produce green and eco-friendly environment.

Iron ore tailings (IOT) are waste material generated when iron ore is processed by separating valuable fraction from the worthless of the ore. Enormous quantities of IOT were produced from mining industries in Malaysia. Although there is no overall statistical data for the quantities of IOT produced in Malaysia but survey of one industry at Kota Tinggi shows that a total 624,000 metric tonnes were produced annually. Majority of these were disposed to landfills due to uneconomic attractive usage. The disposal of waste materials into landfill is not only detrimental to the environmental issues but also decline substantial amount of production profit to the mining industries. Moreover, due to the dwindling of space for landfill and growing restrictions on environment, landfill cost might be high. Besides, Malaysia's strenuous efforts to increase the production of iron ore from 3.5 million tons in 2011 to 10.7 million tons in 2012 for economic development and sustenance for iron and steel industries have increased the IOT disposal across the current 98 iron ore mines scattered throughout Pahang, Johor, Perak and Terengganu (malaysiafactbook, 2013). Therefore, it is expected that more amount of IOT material will be generated from the mining industry and disposed to the environment in near future. The continuous disposal of IOT endures over-burden to the mining industries and the community in terms of environmental and economic perspective. Lack of space for the disposal of huge amount of IOT stocked in the industry will become a major problem in the future. There are possible issues of leaching of heavy metals and acid mine drainage which might cause havoc to the community and the environment (Hitch et al., 2010).

IOT that was stockpiled in the tailing dams might be risky to the environment and the impacts could be physical, chemical or geotechnical instability. The possible effects for storing the tailings in the dam is ground and surface water pollution due to toxic substances such as leads, sulphates and dissolved metals. Sulphates in particular is susceptible to undergo chemical oxidation when exposed to oxygen and form acid in the soil. There are possible issues of leaching of heavy metals and acid mine drainage which might cause havoc to the community and the environment (Hitch *et al.*, 2010)

The persistent disposal of IOT in landfills or tailing dams has range of environmental issues, which include: erosion, dust, water and soil pollution, negative effects on the ecosystems and loss of land fertility. The difficult situations that might be encountered is during the failure of tailing dams or collapse of heaps due to earthquakes and high rainfall, which could affect the environment and health safety of human life (Cai *et al.*, 2011).

In order to find solution to the environmental issues raised, there is need to further study and come up with sustainable utilization of the IOT generated. Current utilization of IOT at 7 to 10% (Huang *et al.*, 2013b; Zhao *et al.*, 2014) is very low compare to huge disposal ranging from 5 to 7 billion tonnes per year worldwide (Edraki *et al.*, 2014). In order to increase IOT utilization, there is need to carry out extensive research on durability and microstructure that will increase the percentage of tailings utilization and clear the suspicion of long-term effect. Such increase in the utilization of IOT will reduce the effects of environmental issues and loss of life of human and aquatic animals. This will provide eco-friendly, economic and environmentally sustainable mining industries and also provide alternative to sand mining for cheaper concrete and construction materials.

### **1.2** Problem Statement

In the past few decades, the demand for construction grade sand is increasing in Malaysia due to rapid economic development and subsequent growth of building activities. This, in many of the occasions has resulted in indiscriminate mining of sand from instream and floodplain areas leading to severe damages to the river basin environment. During the year 2010, Malaysia consumed 2.76 billion metric tons of natural aggregate, out of which 1.17 billion metric tons, or 42.4%, was sand and gravel. The percentage of total aggregate production that is sand and gravel varies widely from state to state. Melacca consumes 7.7% sand and gravel, which is lower than any other state. Selangor, Johor, Terengganu and Federal territory (Kuala Lumpur and Putrajaya) all consume 100% sand and gravel (Ashraf *et al.*, 2011). Sand mining has environmental issues, which include: depletion of virgin deposits, destruction of landscape, reduction of farm and grazing land, collapsing of river banks, deforestation and water pollution (Ako *et al.*, 2014). The remedies for these impacts is the use of waste material as alternatives to river sand (Sreebha and Padmalal, 2011).

On the other hand, the worldwide is clamouring for the conservation of natural raw materials due to increasing demands of construction industry fuelled the intense global research towards economic utilization of the waste to produce eco-friendly construction materials for durable and sustainable concrete structures. Over the period of time, various research works were conducted to engage the use of waste materials from industrial by-products as fine aggregate in concrete structure. IOT materials which were used for aggregate materials among others also played significant roles. Despite its utilization as aggregate material in concrete, the problem of durability remains unsolved. Various researchers agitate for durability test for concrete with IOT. Ugama and Ejeh (2014) suggested that, durability test, varying water-cement ratio and mix design of concrete with IOT should be investigated. Kuranchie et al. (2015) reported that the ferrous content in the IOT has significant negative effect on the corrosion which might have long term effects on durability of concrete. Corrosion of concrete has an important effect on the durability performance of concrete (Güneyisi et al., 2013). Hitch et al. (2010) also reported that waste of mine tailings containing sulphide minerals and heavy metals pose environmental risk due to oxidation and

subsequent production of acid. Hence, the need for long-term durability test is imminent.

With the expansion in the exploration of iron ore to satisfy the iron and steel industry demand, the consequence is the generation of iron ore tailings which are expected to pose further problems. Moreover, to satisfy the future demand and desire needs, further research on durability and utilization of IOT in concrete is required for sustainability and environmentally eco-friendly.

### **1.3** Objectives of the Research

The main aim of this research is to use IOT as a substitute to sand replacement in concrete with the following objectives:

- i). To investigate IOT (physical, mechanical and chemical) properties according to standard requirements for sand (ASTM, BS or EC2).
- ii). To determine the effects of IOT on the properties of fresh and hardened concrete.
- iii). To evaluate the effect of IOT on concrete durability and microstructural properties.

### **1.4** Scope of the Research

The research is experimental in nature, and mainly focused on the development of concrete containing IOT at replacement level of 25%, 50%, 75% and 100% to river sand. Mix proportion of grade 30 and 60 MPa at 0.60 and 0.40 water/cement ratios

were considered. The properties of the constituent concrete materials including leaching behaviour and microstructural characteristics of IOT were examined.

The evaluation of workability, strength properties, deformation (modulus of elasticity) and durability characteristics of concrete were also investigated. This includes the slump, compressive, indirect tensile and flexural strengths, ultrasonic pulse velocity (UPV), drying shrinkage, modulus of elasticity, heat resistance, chloride penetration test, carbonation test, electrical resistivity, resistance to acids and corrosion measurements.

Optimized mixes were used to study the microstructure test in terms of Field Emission Scanning Electron Microscopic (FESEM), X-ray Diffraction analysis (XRD), Fourier Transform Infrared Spectroscopy (FTIR), Thermo gravimetric and differential thermal analysis (TGA/DTA) of hardened concrete.

### **1.5** Significance of the Research

Generally, the use of waste mines like other recycling effort limits wastes disposal (Lottermoser, 2011). At present, these wastes are disposed as landfill materials without any economic benefit in return and considerable amount of money is needed for acquiring land for disposal. The utilization of IOT has become an attractive alternative in construction industry. Thus, using IOT for concrete as a replacement for fine aggregate will slow the consumption of natural resources, encourage innovation and local industries, create jobs and teach responsibility for the environment shared by all.

Finally, when the technology of concrete incorporating IOT is articulated, it will reduce the cost and consumption of natural sand and overall construction cost, thus producing green concrete and make construction affordable.

### **1.6 Research Questions**

This research seeks to address the following questions:

- a) Is IOT physical, mechanical and chemical properties are within the requirements of relevant (ASTM, BS or EC2) codes and standards?
- b) Will IOT contribute in improving strength and other properties of concrete?
- c) Will IOT causes any negative effect on the durability and pore structure?

### **1.7** Thesis Organisation

This thesis is classified into seven different chapters:

**Chapter 1:** This chapter contains a general appraisal and overview of the background problem. It also identified the aim and objectives, scope of the study, significance of research and research questions.

Chapter 2: Review of the available, relevant and related literatures.

**Chapter 3**: The chapter focuses on the breakdown of the experiments for this research involving methodology for characterization of materials used and the procedures for the tests of fresh and hardened properties; and durability issues of concrete.

**Chapter 4:** The chapter analyses and discusses the results of physio-chemical properties of IOT and the effect of IOT on fresh concrete properties. The chapter also discusses the results obtained on workability and hardened properties in terms of

compressive, flexural, tensile strength, ultrasonic pulse velocity (UPV) and modulus of elasticity.

**Chapter 5:** This chapter reports the results and discussions arising from the various durability tests conducted on control specimen and IOT concrete. Aspects of durability test considered in this chapter are; drying shrinkage, water absorption, heat resistance, rapid chloride ion penetration, accelerated carbonation test, electrical resistivity, resistance to acids, accelerated corrosion measurements and electrochemical resistance techniques of the concrete.

**Chapter 6:** Thermo gravimetric analysis (TGA), field emission scanning electron micrograph (FESEM), Fourier transform infrared spectroscopy (FTIR) and X-ray diffraction analysis (XRD) results are analysed and discussed in this chapter. The microstructure studies were examined at 7 and 28 days of concrete strength development.

**Chapter 7:** This chapter deals with the conclusion and recommendations based on the research findings.

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