EARTH-BASED CONSTRUCTION MATERIALS REINFORCED WITH TROPICAL PLANTS AND RECYCLED WASTE CELLULOSE PULP FIBRE: PERFORMANCE AND DURABILITY ASSESSMENT

A thesis presented to the department of

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In Partial Fulfilment of the Requirement of Degree of

Doctor of Philosophy

By

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Abuja, Nigeria

February 2021

CERTIFICATION

This is to certify that the thesis titled "EARTH-BASED CONSTRUCTION MATERIALS REINFORCED WITH TROPICAL PLANTS AND RECYCLED WASTE CELLULOSE PULP FIBRE: PERFORMANCE AND DURABILITY ASSESSMENT" submitted to the school of postgraduate studies, African University of Science and Technology (AUST), Abuja, Nigeria

for the award of the Doctor of Philosophy degree is a record of original research carried out

by

Tido Tiwa Stanislas in the Department of Materials Science and Engineering

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ABSTRACT

Due to the growing concern of deforestation, renewable materials such as recycled cellulosic waste and non-wood fibres provide an alternative solution for partial replacement of wood resources as a reinforcement agent in building blocks. This study consists of exploring the feasibility of producing cellulose pulp and nanofibrillated cellulose (NFC) from plants widespread in Tropical region; raffia fibre (*Raphia vinifera*), cassava bagasse (*Manihot esculenta*), ambarella (*Spondias dulcis*), and bamboo (*Bambusa vulgaris*); to assess their suitability as a source of reinforcing agent for composites. The recycling process of municipal waste fibres was also studied to promote the conservation of plant resources, as well as the circular economy. Bamboo organosolv pulp (BOP) and recycled waste carton pulp (RWCP) were used as a reinforcement phase in an earth-based matrix at varying fibre contents (0, 5, 7.5, and 10%wt.) to assess their performances and durability for civil engineering construction applications. Fibres were produced using both organosolv and soda methods to assess the effect of processing on fibre properties. The composites were manufactured by the extrusion process and tested after 28 days. The results show that the inclusion of RWCP fibre in the soil matrix significantly improved the performance of the composites compared to matrices reinforced with BOP fibres. Addition of 5 %wt. of RWCP, showed an improvement in flexural strength (56%), specific energy (614%), fracture toughness (57%), wear resistance (48%), and thermal insulation (21%) compared to the control sample. The inclusion of RWCP in earth-based matrix increases the moisture loss, the drying shrinkage and behaves as a water reservoir for earth-based materials. It has been concluded from this study that RWCP has the potential to serve as a suitable reinforcement for the promotion of lightweight earthen wall block materials (reduction of bulk density up to 21% after the inclusion of 10% of RWCP), where flexural strength, ductility, and thermal insulation performance are the primary requirements. Besides, the successful replacement of virgin BOP fibres with RWCP fibres reduces the environmental footprint of the building material. Therefore, the use of this RWCP in the construction industry will be an attractive alternative as it will solve both energy and environmental concerns.

DEDICATION

This thesis is dedicated to my wife, my son, my daughter, my mother, my sisters and my brothers for their immeasurable support in my University career.

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I am very grateful to Dr Emeso Beckley Ojo for a helpful technical discussion and useful concepts on the methodology of this work. We also thank the Pan African Materials Institute, the African Development Bank, the African University of Science and Technology of Abuja and the Faculty of Animal Sciences and Food Engineering of the University of Sao Paulo, Brazil for their financial support.

Special thanks to my supervisors, Professor Holmer Savastano Junior and Professor Foba Tendo Josepha for their patience, love, advice and insightful comments. They are parents that I have never had in my entire life and with the respect I owe them. Their contribution to my academic career is immeasurable. Thank you for everything. I would like to express my sincere thanks to the members of my committee (Professor Azikiwe Peter Onwualu, Professor Winston Oluwole Soboyejo and Professor Njeugna Ebenezer) for their insightful comments.

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PREFACE

This dissertation is the original property of Tido Tiwa Stanislas. This work was performed between July 2018 to February 2021 for obtaining the Doctor of Philosophy in Materials Science and Engineering Department at the African University of Science and Technology, Abuja, Nigeria.

I was the principal investigator and this work belongs to me. I was responsible for all major activities including concept formation, experiments, data collection and analysis including the composition of manuscripts, while Prof. Holmer Savastano Junior and Prof. Foba Tendo Josepha were supervising authors involved in the early stages of the concept and composition of the manuscript.

Professor Winston Oluwole Soboyejo, Professor Azikiwe Peter Onwualu and Professor Njeugna Ebenezer were of the committee members and they were actively involved in concept development and manuscript editing.

At the time this thesis was submitted, Chapters 3 and 4 have been accepted for publication in the Journal of Natural Fibres and the Journal of Building Engineering, respectively. Another chapter 5 has been submitted to the Arabian Journal for Science and Engineering for publication.

LIST OF PUBLICATIONS

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Stanislas, Tido Tiwa, Josepha Foba Tendo, Ronaldo S. Teixeira, Emeso B. Ojo, Gbétoglo Charles Komadja, Marzieh Kadivar, and Holmer Savastano Junior. "Effect of Cellulose Pulp Fibres on the Physical, Mechanical, and Thermal Performance of Extruded Earth-based Materials." Journal of Building Engineering (2021): 102259.

Tido T. Stanislas, Gbetoglo Charles Komadja, Odette F. Ngasoh, Ifeyinwa Ijeoma Obianyo, Josepha Foba Tendo, Peter Azikiwe Onwualu, Holmer Savastano Jr. "Performance and Durability of Cellulose Pulp Reinforced Extruded Earth-based Composites." *The Arabian Journal for Science and Engineering* (2020): Under Review.

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CHAPTER ONE

1.0.INTRODUCTION

1.1.Background.

In 2015, the housing deficit in Cameroon was estimated at 1.5 million units and growth at more than 150,000 housing units per year (The World Bank, 2020), 80% of which in urban areas and mainly in the two main cities of the country, Douala and Yaoundé. According to the United Nations, Cameroon's urbanization rate was over 55% (United Nations DESA, 2019) in 2017 and is expected to reach 73% by 2050. Since the cost, availability and properties (strength, thermal insulation and durability) of materials greatly influence the choice, there is a need to explore new ways to produce robust and durable building materials from locally available resources, which can solve both environmental and energy concerns. However, the Minister of Scientific Research and Innovation of Cameroon through MIPROMALO (Mission for the Promotion of Local Materials) promotes the use of local materials to reduce the production cost of national equipment. This research is part of the current objectives of MIPROMALO for the period 2018-2020 and is structured into three sub-programs. The first is to identify, characterize the raw materials, and the second is to develop appropriate technologies in the efficient implementation of building materials and the third is to carry out the extension, training and transfer of technology.

These needs have recently spurred a lot of research into local materials for durability, low cost, high strength and low thermal conductivity for building construction applications (Laibi et al., 2018; Obianyo et al., 2020; Ojo et al., 2020). The earth-based material is cheap, environmentally friendly, and available worldwide for the construction industry. The soil has been used extensively for building walls all over the world, especially in developing countries (Kabiru, 2015). Lateritic soils are abundant in the tropics and cement stabilization trials of lateritic soils have yielded interesting results, but its potential for block making has not yet been satisfactorily explored (Binici et al., 2007). The cost of raw materials for construction in Africa is often exorbitant, especially when most materials are imported. Binici et al. (2007) showed that the straw fibre reinforced concrete brick house is inferior to the mud-brick house in keeping the indoor temperature stationary in winter and summer.

The literature on stabilized earth composites suggests that there is the potential to produce lightweight and strong composites with interesting thermal insulation properties, by controlling fibre content, fibre type/size and production conditions (Chaib et al., 2015; Danso et al., 2015a, 2015b; Kolawole et al., 2017). The addition of natural pozzolan or sawdust in bricks made from lateritic soil tends to maintain their strength while decreasing the density and the thermal conductivity (Meukam et al., 2004). The addition of cellulose pulp fibre in the cement matrix can improve the flexural strength, toughness, and decrease the weight and thermal conductivity of the composite (Correia et al., 2018; Ojo et al., 2019). It is well known that the inclusion of cellulose fibre in the cementitious material improves the mechanical, physical and thermal performance of the building material. However, little work has investigated the influence of using cellulose pulp fibre on the physico-mechanical performance, thermal conductivity, and durability of cement-stabilized extruded earth-based materials. In particular, the study of the effect of the inclusion of cellulose fibres on the post-crack behaviour during bending and shrinkage cracking testing is limited.

This study attempts to develop a building material from recycled waste and virgin pulp to improve resistance to cracking during loading and shrinkage, durability and reduce thermal conductivity properties of the soil matrix. The production of cellulose pulp fibre from lignocellulosic fibre results in improving the specific surface area of the fibre which exhibits a significant improvement in the adhesion force between the fibre and the matrix. This improvement in the interface link between the fibre and the matrix will lead to good charge transfer from the matrix to the fibre.

1.2.Problem statement

Earth-based materials reinforced with cellulose pulp exhibit a remarkable improvement in fibre-matrix bond, greater reinforcement efficiency and a significant increase in flexural strength and toughness compared to using fibres plants in the form of strands (Karade, 2010; Savastano et al., 2003). Wood pulp, which is an example of cellulose fibre, provides a high specific binding area, mechanical anchoring and crack bridging in a brittle matrix due to its high aspect ratio filaments, irregular surface and low coarseness[1] (Savastano et al., 2004). In addition, Figure 1.1 illustrates the crack bridging mechanism of conventional fibre cement composites reinforced with large fibres (macrofibres) (Figure 1.1a) and microfibers (Figure 1.1b) (Robert & Jason, 2017). Macrofibres bridge cracks on a macroscale and absorb energy through plastic deformation, and provide greater toughness to the fibre-cement composite (Figure 1.1a). While microfibers provide a more efficient bridge of cracks on a microscale, preventing these microcracks from growing and coalescing and helping to improve tensile strength and toughness (Figure 1.1b). Thus, the inclusion of microfibers (cellulose fibre) may change the mode of failure

[1] Weight per unit length of pulp fibres

of the earth based matrix from brittle to ductile fracture when subjected to the bending test.

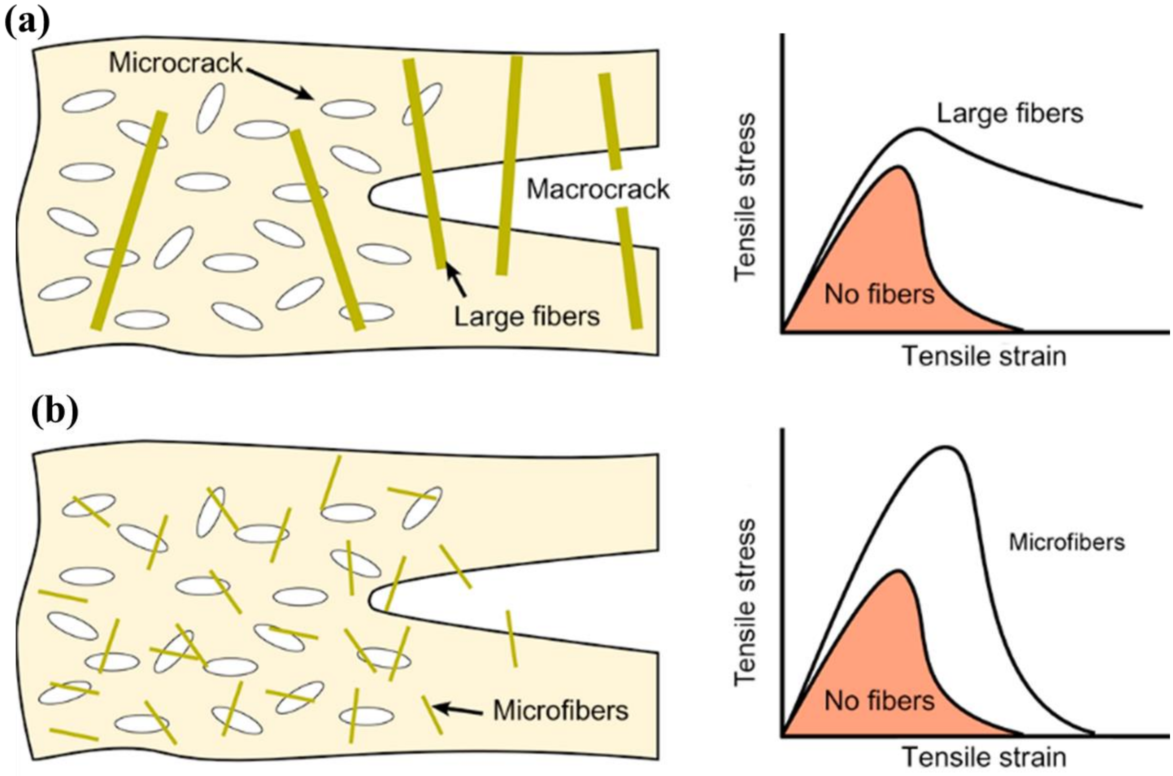

Figure 1. 1 Illustration of the effect of fibre length (a) macrofibres (b) microfibers on the reinforcement mechanism (Robert & Jason, 2017)

With regards to durability in stabilized soil materials, the major concern is the loss of structural integrity when exposed to inclement weather (wind, rainstorm, and other degrading agents) (Manu, 2013; Ojo et al., 2018). The effectiveness of any stabilization mechanism must be examined with a strict durability test to assess the risk of leaching of the binder during service life (Avirneni et al., 2016). This may be due to temperature variation and the infiltration of rainwater into the exposed structure. Considering its sensitivity to weathering, the durability over wetting-drying (w-d) cycles of the cellulose pulp reinforced soil-building material is an important criterion for evaluating the service life. However, studies on this feature to date are very limited.

Shrinkage is considered to be one of the main drawbacks identified in the long-term durability of building materials, as it causes micro and macro-cracks at early ages. (Jiang et al., 2014; Sivakumar & Santhanam, 2007). The mitigation of stresses developed during the drying process and the formation of shrinkage cracks are generally provided by the use of fibres as a reinforcing mechanism to bridge the crack propagation. Several studies have been carried out to understand the limitation of the application of soil material in the building industry and enhance its life span by introducing additive binder (Kampala et al., 2014; Walker, 1995), agricultural waste fibres (Danso et al., 2015b), coconut and sisal fibres (Ghavami et al., 1999), and straw (Bouhicha et al., 2005). The authors agreed that the swelling and shrinkage of natural fibres in drying earth-based material constitutes the major problem of the use of natural fibres in soil matrices. However, few works have addressed the use of cellulose pulp as reinforcement in soil building material to improve the fibre-matrix bond, shrinkage cracks, and the toughness to expand the applications (Muñoz et al., 2020; Ojo et al., 2019).

1.3.Research Objectives

The main objective was to study the reinforcement efficiency of non-wood plants cellulose pulp and recycled waste carton pulp on a stabilized extruded soil matrix, to assess the possibility of replacing virgin fibre with waste from recycled fibre to promote resource conservation and circular economy.

The production of cellulose pulp and nanofibrillated cellulose (NFC) from prevalent Tropical plants; raffia fibre (*Raffia vinifera*), cassava bagasse (*Manihot esculenta*), and ambarella (*Spondias dulcis*), bamboo (Bambusa vulgaris), and municipal waste were studied to explore their feasibility and assess their suitability as a source of reinforcing elements for sustainable building materials.

To improve the physical-mechanical properties and durability of the soil matrix, ordinary Portland cement (OPC) was used as a stabilizer. The effect of the inclusion of cement on the performance of the composite was investigated under different curing conditions while varying the cement content to assess the optimal stabilizing effect of this particular soil.

Two alternative non-conventional fibres (bamboo organosolv pulp and recycled waste carton pulp) were chosen based on their production efficiency to assess the feasibility of cellulose pulp in extruded earth-based materials and to evaluate the possible replacement of virgin cellulose fibre (bamboo organosolv pulp) with recycled waste fibres. This cellulosic fibre is introduced at different percentages in the cement stabilized soil to enhance their flexural strength, shrinkage cracks bridging and thermal insulation performance of the composite.

To assess the dimensional stability, durability, and the mechanism of crack propagation during loading and shrinkage of the earth-based composite reinforced with cellulose fibres, composite samples were subjected to fracture toughness test, repeating drying shrinkage test, and durability against wetting and drying cycles.

1.4.Scope of work

This experimental study evaluated the influence of processing, composition, and size of cellulose fibre on the enhancement of anchorage within the matrix and fibres, physical-mechanical performance, thermal conductivity, and durability of earth-based building material. The production of cellulose pulp fibre reinforced earth-based matrix was done in two stages: (1) production and characterization of cellulosic pulp and nanofibrillated cellulose from raffia fibre and bamboo chips and recycled waste carton,

(2) production and characterization of cement stabilized earth-based composite reinforced with cellulose pulp by extrusion process (Figure 1.2).

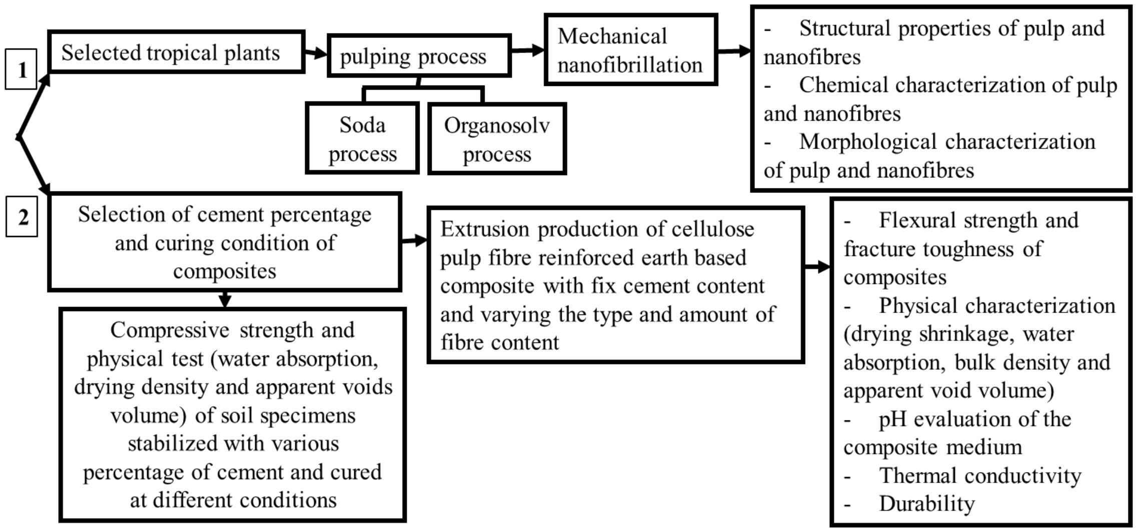

Figure 1. 2 Flowchart of the two stages of evaluation of an earth-based composite reinforced with cellulose fibres

The background to the study and introduction are resented in chapter one. Chapter two presents a current review of the literature on the pulping process, nanofibrillation and fibre reinforced cement stabilized earth composite. The production and characterization of cellulose pulp and nanofibrillated cellulose from tropical plants is discussed in Chapter Three.

In chapter four, the work evaluated the effect of ordinary Portland cement and cellulose pulp fibres on water absorption, bulk density and apparent void volume, mechanical strength, thermal conductivity, and drying shrinkage of earth-based material. The evaluation of the effect of cellulose pulp fibre on the flexural strength, and fracture toughness and durability are presented in chapter five.

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CHAPTER TWO

2.0. LITERATURE REVIEW

2.1. *Lignocellulosic fibres*

Lignocellulosic fibres can be considered as composites of hollow cellulose fibrils held together by a lignin and hemicellulose matrix (Figure 2.1) (Jayaraman, 2003). However, pectin and waxes may be found in lower quantities, while pectin provides the flexibility of the fibre, and the waxes make up the last part of the fibres and alcoholic compounds (John & Thomas, 2008). Lignocellulosic fibres present several interesting advantages particularly their low density, their bio-renewable character and their availability everywhere at modest cost and in a variety of morphologies and aspect ratios, and represents a key source in many different industrial sectors (Fortunati et al., 2019) (Tonoli et al., 2009). The production of cellulose fibres, also at the nanoscale, and their application in composite and nanocomposite approaches, have expanded the attention due to their high strength and stiffness combined with low weight, biodegradability, renewability, and sustainability (Klemm et al., 2011). Compared to cellulose, hemicellulose has a lower molecular weight and has a lower degree of crystallinity. It is also responsible for the swelling behaviour of fibre (Monga, 2017). During the pulping process, the hemicellulose considerable dissolves in the solution; despite that, an essential quantity of hemicellulose is always associated with the pulp (Monga, 2017). Lignin represents an amorphous and aromatic substance and containing phenolic methoxyl, hydroxyl and other constituents groups. It is also called "incrusting material" forming a part of the cell wall and middle lamella in fibre (Monga, 2017). The removal of the lignin compound in lignocellulosic fibre is the main objective of pulping process and therefore, the determination of the amount of lignin in the raw material is essential for choosing the efficient pulping method.

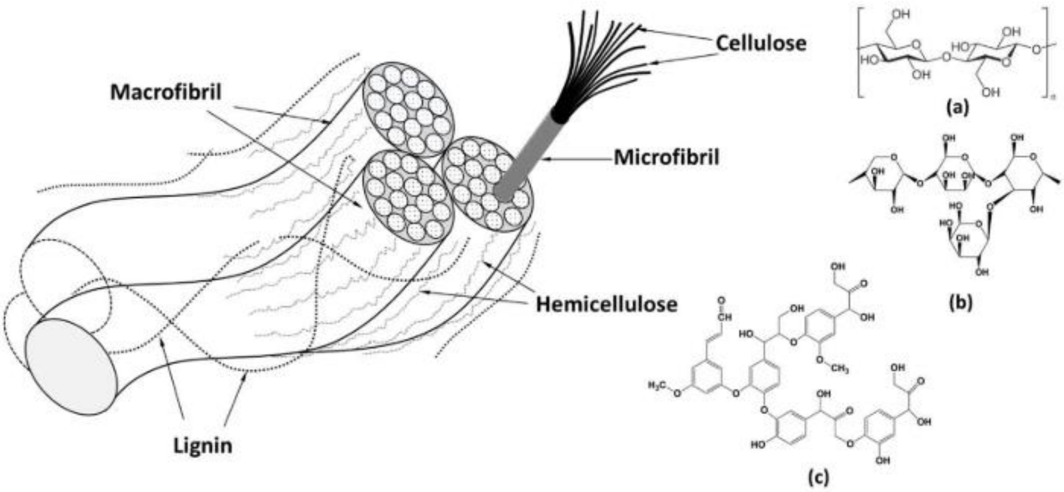


Figure 2. 1 Typical schematic diagram of fibril matrix structure of plant fibre and chemical composition (a) cellulose, (b) hemicellulose and (c) lignin. Source (Gowthaman et al., 2018).

The non-wood lignocellulosic materials account for less than 10% of worldwide pulp production and the most used for cellulose production are distributed in 44% straw, 18% sugarcane bagasse, 14% reed, 13% bamboo, and 11% others (Sridach, 2010).

2.1.1. *Bamboo*

The rapid growth, relatively low cost, and worldwide availability of bamboo materials along with its strength, constitute a promising fibre source for pulping process and reinforcement in various composites as compared to other vegetable sources (Correia et al., 2015). Bamboo can be harvested after 3 to 6 years and the growth rate is estimated at 4.5-7.5 tons/ha**.**y, which is twice that of most fast-growing wood trees (Zhao et al., 2010). Bamboo has high fibre strength, and as one of the major non-wood and available forest resources, it plays an important cultural and economic role in the tropical areas (Correia et al., 2016). For example, bamboo is considerably used in China (around 25% of the world’s bamboo) and the production is estimated at around one million tons (Zhao et al., 2010). Bamboo as a structural material has been intensively analysed, but the use of residue from bamboo (Figure 2.2) to produce particleboard and cellulosic pulp as reinforcement is not well studied. However, the unique growth rate, self-generation, and mechanical performance places bamboo as a competitive material in relation to wood and non-wood lignocellulose material source to produce cellulose pulp (Correia et al., 2015; Coutts & Ni, 1995; Zhao et al., 2010) and cellulose nanofibre (Chen et al., 2011; Chitbanyong et al., 2018; He et al., 2013; Jiao et al., 2016) as reinforcement in an organic and inorganic matrix such as polymers and cement composite. Moreover, the application of bamboo cellulose pulp and nanofibrillated cellulose as reinforcement components in the earth-based matrix has not yet been elucidated in the literature. Therefore, this study attempted to evaluate the effect of bamboo pulp on the physical-mechanical performance and durability of extruded earth-based composite.

Figure 2. 2 Bamboo chip

2.1.2. *Raffia fibre*

Raffia (Raffia bamboo) offers a real opportunity to have affordable building materials to the poor who inhabit the tropical regions because it is a cheap, abundant, and fast-growing material that can meet the need for broad economy housing (Foadieng et al., 2017). The leaves are used for shelter and the stem produces palm sap, which is drunk as a beverage (Obahiagbon & Osagie, 2007). The large numbers of fibres present make raffia a useful raw material (Figure 2.3). It is used for making textiles, cordage, mats and for other purposes including the decoration of masks (Sandy & Bacon, 2001). Raffia is also used in the West Region of Cameroon as a building material especially as ceiling material, but this use is still local (Mbou et al., 2017). Several works have been carried out on the raffia plants to evaluate their insulation properties (James & Tamunoiyowuna, 2016), physical-mechanical performance (Elenga et al., 2009; Tagne et al., 2018; Tagne et al., 2017), and reinforcement efficiency in construction engineering (Elenga et al., 2009). However, the production of cellulose pulp and nanofibrillated cellulose from raffia fibre has not yet highlighted in the literature. Therefore, this work attempted to investigate the behaviour of cellulose pulp and nanofibre produced from raffia fibre for potential application as a reinforcement in stabilized earth bricks.

Figure 2. 3 Raffia fibre

2.1.3. *Cassava bagasse*

African annual production of cassava roots is estimated at approximately 147 million Metric Tonnes (MT), which represent 54% of world production (Ekop et al., 2019). The continuous growth and prospering of cassava processing businesses in most developing countries have resulted in the generation of large amounts of cassava processing wastes and residues (Ekop et al., 2019). Figure 2.4 shows the bagasse extracted from cassava.

The main component of cassava bagasse is water (70-80 %wt.), residual starch and cellulose fibre (Teixeira et al., 2009). The proportion of starch and fibre in cassava bagasse is estimated at the range of 61.84-69.90 % (dry mass) and 10.61-14.35 % (dry mass), respectively (Srinorakutara et al., 2004). This value of natural fibre in cassava bagasse suggest a possibility of using this source lignocellulosic fibre as a raw material for cellulose pulp and nanofibres production.

Figure 2. 4 Cassava bagasse

2.1.4. *Hog plum*

Spondias dulcis is a fast growing equatorial plant with edible fruit which is popular in the name of cassimango in Cameroon and amra in Bangladesh while its English name is hog plum or golden apple (Islam et al., 2013). These are important economic tropical plants that are extensively cultivated in many developing/emerging economies. Hog plum is commonly used as fruit source, the fruit is a remedy to cure itchiness, internal ulceration, sore throat, and skin inflammation, while the astringent back is used to cure diarrhea, eyesight enhancement and eye infection (Islam et al., 2013; Morton & others, 1987; Rahmatullah et al., 2009). However, the piths of hog plum has a lot of fibres (Figure 2.5) and to date, there is little literature on the potential of these plant residues as sources of pulp and nanofibrillated cellulose.

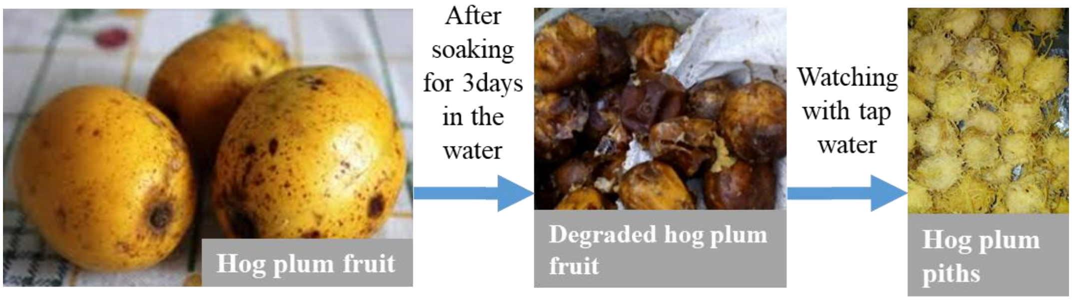


Figure 2. 5 Degradation process of Hog plum fruit after 5 days of immersion in water

Furthermore, several methods have been used to obtain pulp from various sources: alkaline-based (Kraft and soda pulping) (Colodette et al., 2011; Jansson & Brännvall, 2014); acid-based (Lam et al., 2004) and solvent-based (organosolv pulping) (Correia et al., 2015; Ruzene & Gonçalves, 2003; Ruzene et al., 2007). Considerable research effort has been carried out to optimise the production of nanofibrillated cellulose from wood and non-wood raw materials: mechanical treatments (grinding process, high-intensity ultrasonication), chemical treatments (sulphuric acid hydrolysis method), biological treatments and electrospinning methods. The characterization and exploitation of various parts of these plants leaves and residues that could serve as feed for pulp and nanocellulose production are important in finding alternative economic use for this biomass.

2.2. Waste cellulosic fibre

The growth of the world's population and its posterity, especially in the emerging economies, has triggered a dramatic increase in purchasing power alongside with the urbanization of society (Mohan et al., 2016). This population growth is leading to the considerable increase generation of municipal solid waste, which ends up mainly in a landfill and contributes significantly to greenhouse gas emissions (Lelieveld & Crutzen, 1992; Liguori & Faraco, 2016; Mohan et al., 2016). Therefore, the development of sustainable technology is necessary to recycle waste and move to a circular economy (Bhaskar et al., 2016; Ma et al., 2018).

According to the World Bank report (Kaza et al., 2018), the estimation of the municipal solid waste generated in 2016 was 2,010 million tonnes and this is expected to grow to 3,400 million tonnes in 2050. In this report, paper and cardboard represented 17% of global waste (Figure 2.6). Generally, the fibers used for the manufacture of cardboard come from the kraft pulp of eucalyptus and pine. Recycled waste paperboard fibre is a promising reinforcement agent, considering its worldwide abundance and relatively low cost compared to lignocellulosic resources. It might additionally contribute to resource conservation and waste reduction as any other recycled waste. Soroushian & Shah, (1993) reported that the recycled magazine paper fibres can replace up to 50% of the virgin fibres without significant damage to the quality of the fibre cement composites. Currently, the use of cellulose pulp fibres became more attractive as reinforcement in the cement matrix for the construction of roofs, partitions, and non-load bearing walls (Correia et al., 2018). Another strategy to minimize the cost of sustainable building materials may be the use of waste/ recycled pulp as a reinforcement of earth-based walling materials, which only requires water and defibrillator at room temperature. Thus the recycling process is relatively fast and efficient compared to soda and organosolv pulping technique.

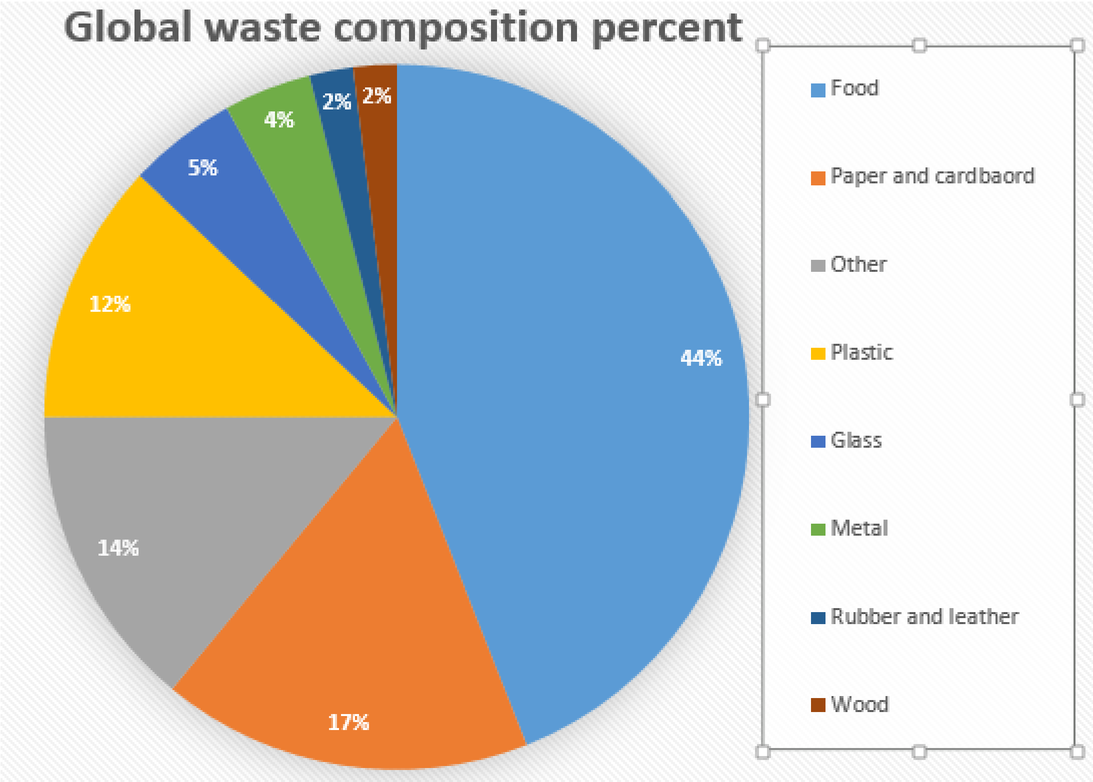


Figure 2. 6 Graph of global waste composition per cent. Source (Kaza et al., 2018)

2.3. Circular economy

The scarcity of materials and the negative environmental impacts such as climate change, the production of waste constitute two major concerns of the traditional linear economy which consists of “take, make and dispose of” (Rathinamoorthy, 2018). In the industrial sector, the linear economy increases risk in all aspects - higher resource costs and supply disruptions due to the scarcity of materials. To minimize the environmental impact, energy flow, improve the lifespan of goods and extend products with available resources and without consuming any extra, Stahel and Reday have developed a new concept called "circular economy" (Stahel, 1994). This innovative concept consists in reducing the pollution generated by waste and consequently in promoting resource conservation strategies by reusing and recycling the product. Figure 4 describes the principle of circular economy in four loops: (1) Reuse, (2) repair, (3) reconditioning and (4) recycling (Stahel, 1982).

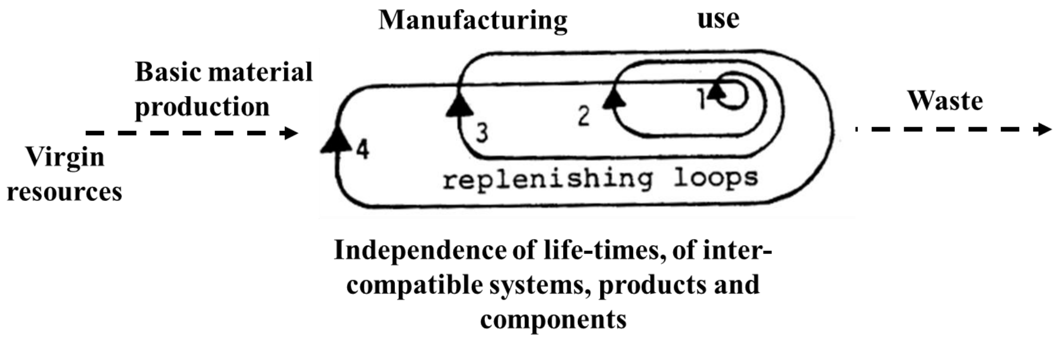


Figure 4: The self-replenishment system (products-life extension) creates an economy based on the reduction of material, energy flow and environmental deterioration without restricting economic growth or social progress and technique (Stahel, 1982).

2.4. *Production of pulp and nanofibrillated cellulose from non-wood vegetable fibres*

The goal of the chemical pulping process is to degrade and dissolve away lignin and leave behind cellulose and hemicellulose in the form of intact fibre. The production of cellulose pulp from non-wood resources had many advantages such as easy pulping and bleaching capacities and excellent fibre (Sridach, 2010). The most used chemical pulping processes are alkaline such as Kraft and those of acidic such as sulphate (Sridach, 2010). Organic solvent solubilize lignin fragment in the organosolv process of pulping, usually associated with water at 10 % to 50% ratio (by volume). There is no need of acid addition for organosolv process taking place at high temperature (185-210 ^{o}C) since the organic acids released from the biomass act as catalysts for the rupture of bonds of the lignin-carbohydrate complexes (Correia et al., 2015). Joaquim et al. (2009) found an optimal condition of producing bamboo organosolv pulp at 190º C for 2 h, 1:1 ethanol/water and 1:10 solid/solvent ratio. The author also achieved a high pulping yield, low lignin content and high mechanical strength. Similar behaviour was reported by (Correia et al., 2015) with bamboo fibre.

Different raw materials such as bamboo, sisal fibre, cassava bagasse, okra fibre, rice husk, hemp, wheat straw, oil palm empty fruit bunch (Correia et al., 2016; Guzman & Egede, 2017; Liu, 2017; Panthapulakkal & Sain, 2013; Rahman et al., 2018; Rosazley et al., 2016; Wicaksono et al., 2013) have been used for the production of nanofibres. Several processes including chemical, mechanical, and physical-chemical treatments have been suggested to-date in the literature to extract nanofibre from lignocellulosic fibre, (Panthapulakkal & Sain, 2013). The mechanical nanofibrillation methods such as ultrasonication, ball milling, high-pressure homogenization, and Masuko milling disc refiner, cause irreversible changes in the fibres and increases the potential for bonding by modifying the morphology and increasing the specific surface of the fibre (Gardner et al., 2008). The commonly used mechanical nanofibrillation process includes Masuko milling disc refiner, which consists of preparing a solution of 2% (w/w) consistency of pulp, then introducing 10 times through the grinder system (supermasscolloider mini, Model MKCA-6-2) at a disc rotation speed of 1700 rpm (Correia et al., 2016).

2.5. *Stabilized earth-based materials*

Earth-based material is a naturally occurring material found in the earth. They include minerals, rocks, and soil and water. This study focusses on clay soil (locally available), which can be used as an alternative building material.

Environmental challenges associated with the production of cement and the challenges of the embodied environmental impact of material has led to growing interest in the use of earth as a construction material (Maskell et al., 2014a; Ojo et al., 2019). However, the greatest single barrier to unfired block construction is the low strength of the material when subjected to high moisture content (Maskell et al., 2014b). To overcome this obstacle, inorganic soil stabilizer: cement, lime, and other hydraulic binders have been used for rammed earth and compressed earth blocks to improve the strength, the dimensional stability and the erosion resistance (Maskell et al., 2014a). The improvement in the performance has been attributed to physical and chemical interactions between the soil, cement and water (Horpibulsuk, 2012). These physical and chemical interactions can be optimized by controlling the process, curing temperatures and moisture conditions (Maskell et al., 2014a).

2.5.1. *Mechanism of stabilization*

There are three main procedures for stabilizing earth materials: mechanical, physical and chemical. Mechanical stabilization involves the compaction of material to increase density and improve strength (Maskell et al., 2014a). Physical stabilization typically means a change in texture and may involve heat and electrical treatment. Chemical stabilization usually means a reaction between stabilizer and soil or stabilizer only. The choice of stabilizer depends on the ability of the additive material to react with the mixing soil. Stabilization of clay soil with cement is an effective and popular technique for improving the shear strength and workability of earth-based materials (Ta'negonbadi & Noorzad, 2017). This work focusses on cement stabilized clay soil.

Calcium-based stabilizers such as Portland cement are summarized in four separate procedures (Prusinski & Bhattacharja, 1998): Cation exchange, Flocculation and agglomeration, Cementitious hydration and pozzolanic reaction.

- Cation exchange

Cation exchange initiates the stabilization process very quickly and is followed by flocculation and agglomeration. The soil plasticity limit is defined as the amount of expansive clay (morillonite) present in the soil. Clay mineral forms by stacking layers of silica tetrahedra and alumina octahedra by ionic and covalent bonds (Prusinski & Bhattacharja, 1998). Due to the negative charge of this crystal structure, cations and water molecules are attracted to its negatively charged surfaces to neutralize the charged deficit (Figure 2.7). This result is the separation of the charged surfaces, forming a diffuse double layer. The thicker this double layer, the more plastic the soil. Monovalent cations such as sodium and potassium are the most prevalent cations which form the double layer, along with water molecules and the soil becomes plastic. To reduce the plasticity of the soil, the monovalent cations present on the surface of the clay must be exchanged in order to reduce the thickness of the double layer. Fortunately, the monovalent cations in the double layer can be easily exchanged for other cations. Cement, which is a good calcium-based soil modifier, can provide enough calcium ions to replace monovalent cations on the surfaces of clay particles. This ion exchange process occurs within hours, shrinks the water layer between the clay particles and reduces the plasticity of the soil. The Principle is illustrated in Figure 5.

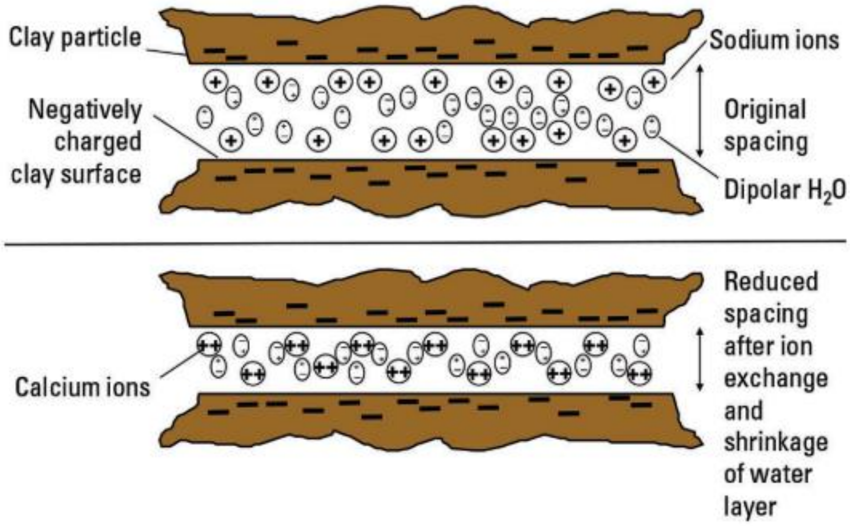


Figure 2. 7 Illustration of cation exchange phenomenon. Source (Halsted et al. 2008)

- Flocculation and agglomeration

Clay soils are light, difficult to handle, and have high void rates. The addition of cement causes flocculation and agglomeration of the soil (Jerod & Wayne, 2020). Flocculation and agglomeration change the texture of the material from that of fine-grained plastic material to one resembling crumbly and granular soil (Halsted et al. 2008). Before the soil undergoes flocculation and agglomeration, clay particles are naturally aligned parallel to each other in layers due to their chemical makeup. After undergoing flocculation and agglomeration, the clay particles are randomly aligned in an edge-to-face orientation, giving the soil a granular-like texture (Figure 2.8). The high electrolyte content and the high pH of the treated soil and the reduction in the thickness of the double layer are all attributes of the dispersion.

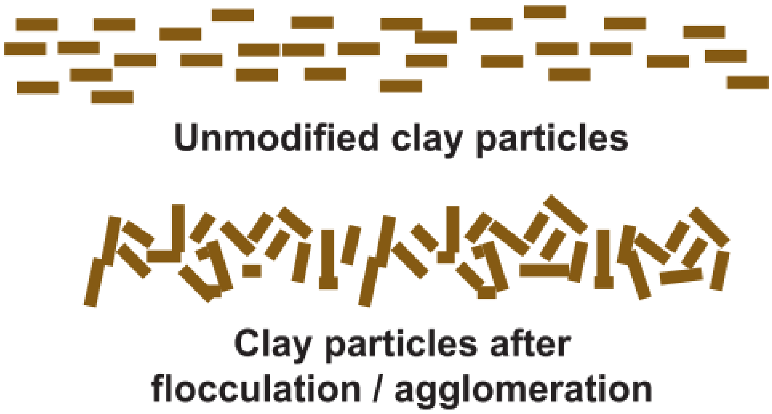


Figure 2. 8 Illustration of flocculation and agglomeration phenomenon. Source (Halsted et al. 2008)

- Cementitious hydration

Portland cement consists of five main compounds such as tricalcium silicate (C_3S), dicalcium silicate (C_2S), tricalcium aluminate, tetracalcium aluminoferrite and gypsum, and some minor compounds (Steven et al., 2002). In the presence of water, the compounds undergo a hydration mechanism as shown in Table 2.1, which results in the formation of calcium hydroxide, calcium silicate hydrate gel (C-S-H), and calcium aluminate hydrate gel (C-A-H) (Kashef-Haghighi et al., 2015). However, gypsum (calcium sulphate) undergoes hydration and forms ettringite ($3CaO.Al_2O_3.3CaSO_4.32H_2O$, or Tri-sulfate), as shown in Table 2.1. The calcium hydroxide compound produced from the hydration of C_3S and C_2S is consumed in the reaction with tetracalcium aluminoferrite ($4CaO.Al_2O_3.Fe_2O_3$ or C_4AF) and gypsum (Table 2.1). The C-S-H and C-A-H act as the “glue” which provides structure in cement modified soil by stabilizing the flocculating clay particles by the formation of strong cement-clay bonds (Figure 2.9). This bond between the hydrating cement and the clay particles improves the gradation of the modified clay by forming larger, aggregate-like particles from fine-grained particles (Prusinski & Bhattacharja, 1998). The majority of this reaction occurs within the first 30 days after adding the cement to the soil.

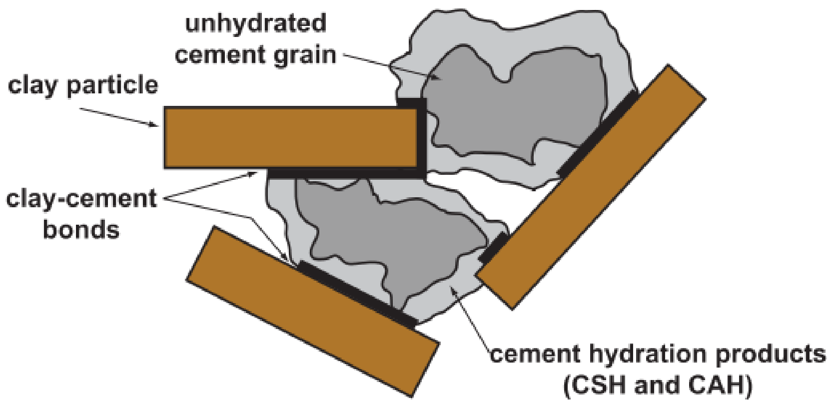


Figure 2. 9 Illustration of the cementitious hydration process. Source (Halsted et al. 2008)

Table 2. 1 Portland cement compound hydration reaction (Steven et al., 2002)

2(3CaO.SiO_2) Tricalcium silicate	+11 H_2O Water	=3CaO.2SiO_2.8H_2O Calcium silicate hydrate (C-S-H)	+ 3(CaO.H_2O) Calcium hydroxide
2(2CaO.SiO_2) Dicalcium silicate	+9 H_2O Water	=3CaO.2SiO_2.8H_2O Calcium silicate hydrate (C-S-H)	+ CaO.H_2O Calcium hydroxide
3CaO.Al_2O_3 Tricalcium Aluminate	+3(CaO.SO_3.2H_2O) Gypsum	+ 26 H_2O Water	= 6CaO.Al_2O_3. 3SO_3.32H_2O Ettringite
2(3CaO.Al_2O_3) Tricalcium Aluminate	+ 6CaO.Al_2O_3. 3SO_3.32H_2O Ettringite	+ 4 H_2O Water	= 3(4CaO.Al_2O_3.SO_3.12H_2O) Calcium monosulfoaluminate
3CaO.Al_2O_3 Tricalcium Aluminate	+ CaO.H_2O Calcium hydroxide	+ 12 H_2O Water	= 4CaO.Al_2O_3.13H_2O Tetracalcium aluminate hydrate
4CaO.Al_2O_3.Fe_2O_3 Tetracalcium Aluminoferrite	+ 10 H_2O Water	+ 3(CaO.H_2O) Calcium hydroxide	= 6CaO.Al_2O_3.Fe_2O_3.12H_2O Calcium aluminoferrite hydrate

*This table illustrates only major transformations and not several minor transformations. The composition of calcium silicate hydrate is not stoichiometric

- Pozzolanic reaction

While cementitious hydration is the primary reaction between cement and water, secondary reactions, known as pozzolanic reactions, also occur (Figure 2.10). These reactions are created from the combination of calcium ion, silica, and alumina. Although pozzolanic reactions occur via a through-solution process, it has been claimed that they are direct reactions between calcium hydroxide ($Ca(OH)_2$) and adjacent clay surface, with the pozzolanic products formed as precipitates (Prusinski & Bhattacharja, 1998). These reactions can be described as follows.

$$Ca(OH)_2 + SiO_2 \rightarrow Calcium - Silicate - Hydrate \quad \text{(eq1)}$$

$$Ca(OH)_2 + Al_2O_3 \rightarrow Calcium - Aluminate - Hydrate \quad \text{(eq2)}$$

Calcium hydroxide results from hydration, which promotes the cementing action. The process takes calcium ions (from cement) and combines them with silica and alumina (clay) to form additional aluminates and silicates. Although pozzolanic reactions occur to a much lesser degree than cement-based hydration, they add more strength and durability to the soil and can last for several months or years. (Jerod & Wayne, 2020).

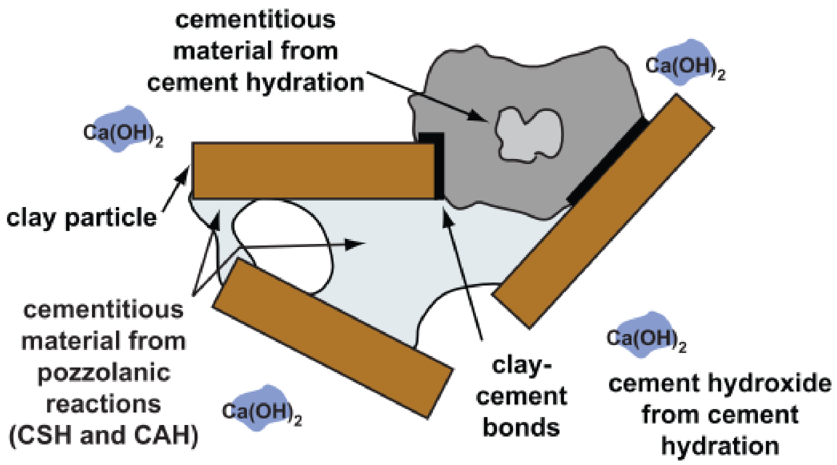


Figure 2. 10 Illustration of pozzolanic reaction. Source (Halsted et al. 2008)

2.6. Natural fibre reinforced earth-based composites

2.6.1. Fibre-reinforced composite

Generally, improvement in the mechanical performance of materials is achieved by two engineering approaches (Soboyejo, 2003). The first approach consists of modifying the internal structure of the material via processing or heat treatment. However, after a certain number of iterations, an asymptotic limit will soon be reached by this approach, as the properties approach the intrinsic limits of a given system. On the other hand, the second approach involves extrinsic modification by the introduction of additional (external) phases with the potential to improve the mechanical properties of the material at the infinite array. For example, the introduction of fibers (external phase) in the earth-based material (matrix) helps to modify the mechanical performance of the material obtained (composite).

A composite material is obtained by combining two or more materials with chemically distinct and insoluble phases to give a unique combination of properties. One of the most unique composites is a fibre reinforced matrix because their constituents are separate and different at the molecular level. The final properties of the composite are often superior to the properties of the constituent materials (Raton et al., 2002). The inclusion of the fibrous phase in the matrix provides higher strength and stiffness to the composite, while the matrix provides stiffness and environmental resistance. However, the properties of the composite are highly dependent on the way the fibres are deposited in the matrix, the dispersion of the fibre and the interface between fibre and matrix (Texeira et al., 2018). This experimental investigation focuses on cellulose fibre-reinforced earth-based composites.

2.6.2. Fibre-reinforced earth-based materials

Historically, the most common method used to minimise shrinkage cracking during the drying process and to improve mechanical strength has been the introduction of fibres in soil matrix (Ojo et al., 2019). In India, Greece and other countries, straws mixed with clay soil to make bricks were used to build houses around 3,500 years ago (Bentur & Mindess, 2006). At the same period, straw-reinforced sun-baked bricks were used to build the 57m-high Aqar Quf hill (near present-day Baghdad) (Bentur & Mindess, 2006). Lignocellulosic fibres as a reinforcement in earth-based blocks have been extensively studied these recent years to improve the compression, flexural strength, tensile strength and durability of building materials (Danso et al. 2015b; Ghavami et al., 1999; Kolawole et al., 2017; Mustapha et al., 2016a; Ojo et al., 2020). Advantages of lignocellulosic fibres compared to other reinforcing fibres are their availability, non-toxic character, low cost, and renewability (Soares et al., 2019). A wide range of natural fibre such as bamboo (Kolawole et al., 2017), sisal (Ojo et al., 2020), coconut husk (Danso et al. 2015), straw (Mustapha et al. 2016a), agricultural waste (Danso et al. 2015b) has been used to reinforce earth-based blocks to improve their bending strength and fracture toughness and durability. This improvement of the fracture toughness in soil composite was attributed to the crack bridging mechanism.

Studies have been carried out on the effect of fibre aspect ratio on mechanical properties of soil building blocks (Danso et al. 2015a), some of which are: the study of the influence of fibres on flexural properties and failure mechanism of compressed soil blocks (Donkor & Obonyo, 2016), the study of strength and fracture toughness of earth-based natural fibre-reinforced composites were evaluated (Mustapha et al., 2016a). Furthermore, the studies were also conducted to evaluate the toughening behaviour in natural fibre-reinforced earth-based composites (Mustapha et al., 2016b). The pull-out behaviour of natural fibre from the earth-based matrix was also evaluated and the results show that increasing the fibre embedment length and the fibre volume fraction (in the earth/cement matrix) increases the peak pull-out load (Mustapha et al., 2016). Mostafa & Uddin, (2016) did the experimental analysis of Compressed Earth Block (CEB) with banana fibres resisting flexural and compression forces. The results show a significant improvement of the compressive strength and the flexural strength at the fibre length of 60 and 70 mm. The authors also recorded an increase in of the toughness of the CEB due to the addition of the banana fibre. However, the influence of cellulose pulp fibre inclusion on the physical, mechanical, and thermal performance of cement stabilised earth-based materials is not well known.

2.6.3. *Cellulose pulp fibres as a reinforcing element in an inorganic composite*

Over the past three decades, considerable research has been undertaken to find an alternative fibre to replace asbestos in fibre cement products and improve the environmental quality of products. This need for new green materials has led to the use of cellulose fibre-based composites. This is because they are available, affordable, have better mechanical properties and require low production energy consumption. (Moshi et al., 2019). Cellulose fibres are classified as natural fibres and are obtained from the processing of wood or natural fibre. In most developed countries, wood fibre-reinforced cement (WFRC) products are well-known and are used commercially with high acceptance for construction purposes because the reinforcement offers improved strength and toughness (Swamy, 1990). Nowadays, cellulose pulp reinforced cementitious matrix is widely used in the production of corrugated and flat sheets, roofing tiles, partition board (walls), and other materials for construction (Soares et al., 2019). However, the use of cellulose pulp fibre as reinforcement in earth-based material are not intensively investigated. Recently, Schweig et al. (2018) studied the mechanical properties of cellulose fibres obtained from recycling Kraft paper reinforced earth-based blocks. The results show slight shrinkage and increased compressive strength in the cracked state, increased toughness and good durability performance. Ojo et al. (2019) studied the effect of eucalyptus pulp on the physical and mechanical performances of alkali-activated clay materials. The results show that the addition of 0.5, 1 and 2 % of eucalyptus pulp volume ratio in the earthen composite provides no significant improvement to brittle failure in the flexural test. This outcome is in agreement with the finding of other studies that reported the efficiency of cellulose pulp fibre reinforced cementitious matrix with the fibre content ranging from 6 to 10 % (Correia et al., 2014; Joaquim et al., 2009; Tonoli et al., 2009). The authors concluded that 2% of the volume ratio of eucalyptus pulp is not sufficient to provide a beneficial contribution to the earthen matrix. In a related study, Muñoz et al., (2020) evaluated the effect of paper and pulp waste on the compressive strength, thermal conductivity and toxicity of adobe bricks by varying the percentage of fibres up to 20%. The results reported an improvement in compressive strength of up to 190%, while thermal conductivity was reduced by up to 30% for 12.5% of the cellulose pulp content. In addition, the bending strength, toughness, pH value of the matrix environment, durability, and drying shrinkage of the earthen wall material reinforced with cellulose pulp fibres should be evaluated to promote and vulgarize the application of this material as building components.

2.6.4. Extrusion process on the properties of fibre reinforced earth-based composites

The improvement of stabilized soil performance has been attributed to physical-chemical interactions between soil, cement, and water (Horpibulsuk, 2012). These physical-chemical interactions can be optimized by controlling the process, curing temperatures and moisture conditions (Maskell et al., 2014a). Although compression moulding is the traditional processing technique for producing stabilized block, the extrusion moulding has been successfully used as an efficient processing method in the forming of earth-based pastes for the production of clay bricks (Ojo et al., 2019). Khelifi et al., (2016) demonstrated that the fibres do not significantly improve the mechanical behaviour of cast materials, but in contrast, the extrusion process improves the fibre dispersion and fibre/matrix bond quality and consequently, enhances the mechanical performance of the composite. Ojo et al., (2019) investigated the effect of fibre reinforcement on the properties of extruded alkali-activated clay soil and revealed the improvement of the mechanical performance of the extruded composite as a result of the high packing density due to the extrusion process.

2.7. Characterization techniques

2.7.1. Physical characterization of earth-based composites

The moisture sensitivity of fibre-reinforced unfired earth materials limits the application of this class of materials to humidity-controlled environments (Ojo et al., 2019). According to ASTM C62 (ASTM, 1989), the maximum water absorption value for severe and moderate weathering resistance (5h boiling test) of the bricks should be 17 % and 22 % respectively. Therefore, the development of the fibre reinforced composite requires an evaluation of the physical behaviour to define a potential application of the material. For instance, Ojo et al., (2019), studied the effect of fibre inclusion on the performance of alkali-activated earth-based material. The results show that the sisal fibre improves the packing density and reduces the water absorption of the composite, while the addition of pulp and polypropylene fibre reduces the density and increases the water absorption capacity of the composite. This increase in water absorption with the introduction of pulp and polypropylene fibre is consistent with the basis of the literature (Danso et al. 2015; Kolawole et al., 2017), which is linked to the presence of free hydrogen groups at the surface of cellulosic fibres, which bind to water. However, the reduction of water absorption with the inclusion of sisal fibre may be related to in situ treatment of the fibre by the alkali-activated matrix, which reduces the water transfer between the fibre and the matrix (Segetin et al., 2007). In this study, the bulk density (BD), water absorption (WA) and apparent void volume (AVV) is obtained from the average of six samples of each formulation by following the procedures specified by the ASTM C 948 Standards (Testing & Materials, 2009). The physical test is carried out after 28 curing days and the properties are calculated using equation (5.1-7.1).

$$Water\ absorption\ (WA) = \left(\frac{M_{sat}-M_{dry}}{M_{dry}}\right) \times 100 \qquad \text{(Eq 5.1)}$$

$$Bulk\ density\ (BD) = \left(\frac{M_{dry}}{M_{sat}-M_{i}}\right) \times \rho \qquad \text{(Eq 6.1)}$$

$$Water\ absorption\ (WA) = \left(\frac{M_{sat}-M_{dry}}{M_{sat}-M_{i}}\right) \times 100 \qquad \text{(Eq 7.1)}$$

Where M_{sat} is the saturated specimen's mass (g) with a dry surface, M_{dry} is the dry specimen's mass (g) after 24h at 105°C, M_i is the immersed specimen's mass in water (g) and ρ is the bulk density of water (g/cm^3).

The drying shrinkage which is defined as the reduction in volume caused primarily by water loss during the drying process can be due to the evaporation of water in the material and represents nearly 70% of the total shrinkage of the cemented material. According to Souza, the variation in the surface tension of colloidal particles, the variation in the shutdown pressure, and the loss of interlaminar water from the hydration of cement constitute the main mechanisms of drying shrinkage (De Souza, 2014). According to (Ghavami et al., 1999) natural fibre is prone to absorb water and swell during the mixture of fibre-reinforced soil composite (Figure 2.11). This expansion disperses the matrix at the micro-scale, and after the drying procedure, the fibres loses the moisture, shrinking to their original size, and leave very small cavities nearby them).

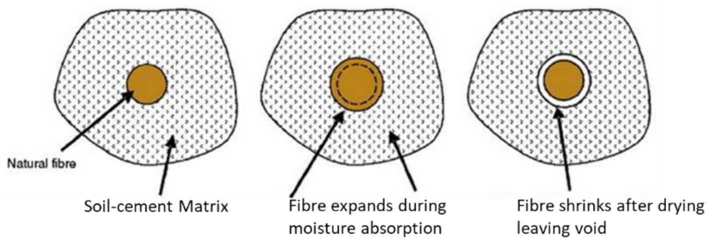


Figure 2. 11 Interaction of natural reinforcing fibre and drying soil (Ghavami et al., 1999).

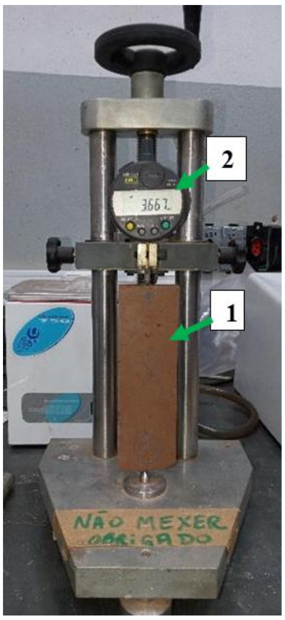
The evaluation of the effect of fibres inclusion on the drying shrinkage is carried out according to the Australian Standards AS1012.13 (Australian Standards, 1992) with a little modification. To ensure a minimum variation in the point of contact with the dial indicator pins and the support, the stainless steel discs with concave fit were glued with epoxy glue to the ends of the specimen (160mm×50mm×15mm). The measurements of the length and moisture variation are carried out with a digital dial indicator from Mitutoyo, Japan with an accuracy of 1 μm and a digital balance Ohaus adventurer, Germany with an accuracy of 0.01 g respectively. Figure 2.12 presents the experimental setup adapted for the measurement. After 28 days of curing, the specimens were directly immersed underwater for 24 h before the first reference measurement. The first measurement of the specimens saturated with water is considered as the reference for the calculation of the shrinkage during drying using equation (8.1). Then the specimens are placed in the controlling environmental chamber with the parameters adjusted to (23 ± 2) °C and (50 ± 5) % RH and measurements are taken daily to verify the variation in length due to the shrinkage of the drying composite for 7 days. Moisture loss ΔW is after drying time t is determined using equation (9.1).

$$D_s = \left(\frac{C_0 - C_i}{l_0}\right) \times 100 \qquad \text{(Eq 8.1)}$$

where D_s is the drying shrinkage (%), C_0 is the initial (reference) length (mm) of the saturated specimen, C_i is the daily measurement of the specimen length (mm) maintaining in the controlling chamber, and l_0 the length of the specimen measured in mm.

$$\Delta W = \frac{W_0 - W_t}{W_0} \times 100 \qquad \text{(Eq 9.1)}$$

where W_o and W_t were the initial (reference) moisture content (g) and moisture content (g) after drying time t, respectively.



1: Composite specimen

2: digital dial indicator

Figure 2. 12 Set-up of the drying shrinkage measurement

2.7.2. Strength and fracture toughness of earth-based composites

One of the main concerns in the production of building materials is to meet the service requirements predicted by stress analyzes. This necessarily implies an understanding of the relationships between the microstructure (i.e. internal characteristics) of materials and their mechanical properties. Most standards of compressive earth-block (CEB) show that the compressive strength is a significant parameter to design constructions. However, the flexural strength and fracture toughness performance of fibre-reinforced earth-based composite should be known to expand the application of this sustainable and ecofriendly material (Kolawole et al., 2017; Mustapha et al., 2016c; Ojo et al., 2020).

Maskell et al., (2014a) study the mechanical performance under dry and wet conditions of stabilized inorganic extruded earth masonry. According to the author, successful stabilization is obtained when the compressive strength is greater than 1.0 MPa in the saturated state and then 3.2 MPa in the dry state. Danso et al. (2015b), evaluated the compressive strength of soil building blocks reinforced with natural fibres from agricultural waste. The results show that the inclusion of agricultural waste fibres in the soil blocks improved the compressive and tensile strength of the blocks with an increase in strength between 16% and 57%. Blocks reinforced with coconut and oil palm fibres were more effective than soil blocks reinforced with bagasse. In the related study, Mustapha et al., (2016a), study the cement stabilized lateritic soil reinforced with straw fibre. The results showed that the compressive strength value was decreasing with the introduction of straw fibre, while there is an increase of flexural strength (up to 8.99MPa) and fracture toughness (up to 1.41 MPa.m$^{0.5}$). Later, Kolawole et al., (2017), evaluated the effect of bamboo fibre inclusion on the compressive, flexural strength and fracture toughness of the cement stabilized lateritic soil. According to the authors, the addition of bamboo fibre improves the compressive strength, flexural strength, and fracture toughness of the composite. Ojo et al., (2019), study the effect of three types of fibre (eucalyptus pulp, sisal fibre and polypropylene fibre) on the flexural strength of alkali-activated clay soil by varying the fibre content from 0.5 to 2% per volume. The results show that the sisal fibre reinforcements gave the most remarkable result with the highest significant enhancement in flexural strength (79% compared to plane specimen) compared to composites reinforced with eucalyptus pulp micro-fibres. The maximum modulus of rupture of 5.5 MPa was achieved with the inclusion of 1 % vol. of sisal fibre in the earth-based matrix. Recently, Muñoz et al., (2020), evaluated the feasibility of reinforced adobe blocks which paper and pulp waste u to 20%. The result shows that the minimum of 12.5% of cellulose fibre is feasible to a reinforced earth-based matrix and the 10% of fibre satisfactory improve the properties of the composite with the increasing of the compressive strength at 25% and decreasing of the thermal conductivity at 50%, compared to unreinforced composite.

2.7.3. *The durability of the earth-based material*

Historically, earth-based building materials are generally stabilized for two reasons: to increase the cohesion and strength of soils that would otherwise be unsuitable for construction and to improve the material's resistance to water-induced erosion (durability). In recent years, several researchers in the construction industry have focused on the development of alternative building material from agricultural waste (bagasse, coconut fibre, oil palm) and non-woody fibres such as bamboo to provide strong, lightweight, environmentally friendly and economically sustainable building materials.

For instance, Danso et al. (2015b) study the effect of agricultural waste fibres on the resistance to wear and erosion of soil blocks. The results show that the inclusion of fibres improves the wear and erosion resistance of earth blocks and the optimum enhancement was achieved with the inclusion of 0.5%wt. of agricultural waste fibre in the soil matrix. Thanushan et al., (2019) evaluated the effect of wetting and drying cycles on the compressive strength of coconut fibre-reinforced earth-based blocks. The result shows that the compressive strength of blocks was decreasing with the increase of the wet-dry cycles, and after twelve wet-dry cycles, the maximum reduction was reported at 22% for coconut fibre-reinforced blocks and 25% for the unreinforced block. According to the authors, this enhancement of durability can be related to the capacity of coconut fibre to absorb tension.

The degradation study against weather of earth-based materials is generally assessed through wetting and drying tests, according to ASTM D 559-57 (ASTM, 1996). This wet and dry cycle helps as a first approach to the material durability. The test consists of immersing the soil sample in water for 5 hours and then taking it out and drying it for 42 hours at 71 ° C. The procedure is repeated for 12 cycles, the samples are brushed each cycle to remove the fragment of the material affected by the wetting and drying cycles.

2.7.4. *Thermal conductivity*

The thermal conductivity of building blocks is an important parameter that aids in the understanding of heat transfer across the material. The material with low thermal conductivity value provides comfort inside the building even in hot and cold countries (Bentchikou et al., 2012). Therefore, more attention should be paid to energy saving, namely thermal conductivity which is a measure of thermal insulating efficiency of the block to provide considerable comfort inside the house. Due to their porous structure (presence of lumen) and their low density, lignocellulosic fibre offers a lower value of thermal conductivity in the cementitious and earth-based matrix and constitutes an alternative solution for energy saving in the construction industry (Bentchikou et al., 2012; Guérin et al., 2018). Mekhermeche et al., (2016), studied the thermal properties of clay bricks reinforced with date palm fibre by varying the fibre content at 1, 2 and 3% by mass. The results reported that increasing the mass fraction of fibres gives rise to a decrease in thermal conductivity, heat capacity, specific heat and thermal diffusivity, while thermal resistance increases. Also, Binici et al., (2007) evaluated the thermal insulation properties of straw fibres reinforced mud brick as wall materials. The result shows that straw fibre reinforced concrete bricks house prove to be inferior to the mud-bricks house in keeping interior temperature stationary during winter and summer. Recently, Muñoz et al., (2020) studied the influence of paper and pulp wastes on the thermal properties of adobe blocks, varying the percentage of fibers up to 20%. The result shows that the thermal conductivity value decreased with the inclusion of cellulose fibre and the minimum value was 0.603 W / (m.K) for 12.5% fiber, which corresponds to 30% reduction, compared to the control sample.

Several published results in literature have found a positive effect of the inclusion of fibers on the compressive strength, flexural strength, tensile strength, density, thermal conductivity and durability of earth blocks. Table 1 presents the properties of soils and natural fibres used in adobes, compressed earth blocks, extruded earth blocks and stabilized earth blocks. Although considerable work has been done on evaluating the properties of fibre-reinforced earth-based materials, few studies have investigated durability against wet-dry cycles, post-peak bending test, fracture toughness and dimensional stability of building block materials (Table 2.1). Furthermore, the evaluation of the reinforcing effects of the cellulose pulp reinforcing earth block is limited. This investigation attempted to produce viable cellulose pulp-reinforced earth-based walling material using non-conventional bamboo organosolv pulp and recycled waste carton pulp. The main objective was to study the reinforcement efficiency of bamboo cellulose pulp and recycled waste carton pulp on a stabilized extruded soil matrix, in order to assess the possibility of replacing virgin fibre with waste from recycled fibre to promote resource conservation.

Table 2. 2 Properties of soils and natural fibres used in adobes, compressed earth blocks (CEB), and stabilized earth blocks (SEB) (Thanushan et al., 2019)

Reference	Block type	fibre			Properties checked						
		Type	Fraction	Length	Density	Compression		Flexural		Fracture toughness	Durability checked
			(%)			peak	post-peak	peak	Post-peak		
Bouhicha Aouissi and Kenal. (2005)	CEB	Straw fibres	0, 1, 1.5, 2	10-500		**X**		**X**			Water absorption and shrinkage
Quagliarini and Lenci. (2010)	Adobe	Straw fibres	0, 0.25, 0.50, 0.75	50		**X**					
Chee-Ming. 2011	SEB	Oil palm fibres	0, 0.25, 0.50, 0.75	10	**X**	**X**					Water absorption
Aymerich, Fenu, and Meloni. (2012)	CEB	Wool fibre	2, 3	10, 20, 30				**X**	**X**		
Gullu and Khudir. (2014)	SEB	Jute fibre	0, 0.25, 0.50, 0.75, 1.0	10, 20		**X**	**X**				Freeze-thaw
Milogo et al. (2014)	Adobe	Hibiscus cannabinus	0, 0.2, 0.4, 0.6, 0.8	30	**X**	**X**		**X**			Water absorption, water spray test, Thermal conductivity
Taallah et al. (2014)	CEB	Date palm	0, 0.05, 0.1, 0.15, 0.2	20-35		**X**					Water absorption
Danso et al. (2015)	Adobe	Bagasse, coconut fibre and oil palm	0.25, 0.50, 0.75, 1.00	38-80	**X**	**X**		**X**			Water absorption, water spray test

Sharma, Vinayak, and Marwaha. (2015)	Adobe, SEB	pinus roxburghu, Grewia optivia	0, 0.5, 1.0, 1.5, 2.0	30		**X**			
Mostafa and Uddin. (2016)	CEB	Banana	0-5	50-100		**X**	**X**		
Zak et al. (2016)	SEB	Flex, hemp	1, 3			**X**			
Kabiru et al. (2016)	SEB	Straw fibres	5, 10, 20	10		**X**	**X**	**X**	
Kolawole et al. (2017)	SEB	Bamboo fibre	5, 10, 15, 20, 25		**X**	**X**	**X**	**X**	Water absorption
Munoz et al. (2020)	Adobe	paper and pulp wastes	0, 2.5, 5, 7.5, 10, 12.5, 15, 17.5, 20		**X**	**X**			Thermal conductivity, toxicity and water resistance

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CHAPTER THREE

3.0.Production and characterization of pulp and nanofibrillated cellulose from selected tropical plants and municipal wastes[2]

3.1.Introduction

The interest in the use of lignocellulosic fibres as reinforcing agents for both polymer and inorganic matrices has advanced remarkably in recent years due to attractive features such as a low cost and density, high specific strength and rigidity, and ease of processing due to their non-abrasive nature (Jonoobi et al., 2009). The industrial application of nano-cellulose presents new opportunities for the valorisation of plant-based biomass residues, especially in tropical countries where forestry, food and cash crop production are important economic activities that generate a diverse range of residues. The response of biomass to pre-treatment and transformation processes such as pulping, bleaching and fibrillation is however highly variable, as a function of morphology and composition and there is a need to characterise each source to generate a database that can be used for process design and optimisation (Chen et al., 2019). This is especially pertinent in a blended feedstock scenario.

Wood and non-wood biomasses serve as starting material for the production of cellulose pulps. However, growing concerns associated with the negative effect of deforestation has stimulated researchers to focus more on agricultural, biomass sources, municipal wastes, and wood discarded by logging within the frame of sustainable forest

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management (Sillero et al., 2020) for pulp production. Furthermore, in developing countries, there is a demand for alternative raw materials, such as natural fibres and binders to substitute synthetics, which are expensive (Raja et al., 2020).

Raffia (raffia bamboo) offers a real opportunity to affordable building materials for low-income earners who inhabit the tropical regions because it is a cheap, abundant, and fast-growing material that can meet the need for broad economy housing (Foadieng et al., 2017). In addition, annual production of cassava roots in Africa is estimated at approximately 147 million tonne and the continuous growth and abundant of cassava processing businesses in most developing countries resulted in the generation of large amounts of wastes and residues (Ekop et al., 2019). *Spondias dulcis* which is a fast-growing equatorial plant with edible fruit which is popular in the name of cassimango in Cameroon and amra in Bangladesh and its English name is ambarella (Islam et al., 2013), these are important economic tropical plants that are extensively cultivated in many developing/emerging economies (Colodette et al., 2011). However, literature is scarce on the potential of these plant residues as sources of pulp and nanofibrillated cellulose. It is well-known that exceptional mechanical performances of bamboo mainly originate from its fibre components (Lo et al., 2004; Yu et al., 2011). The application of bamboo cellulose fibres as reinforcement phase to enhance the mechanical performance of the composites have been attracted by several researchers in the field of materials science. Nowadays, one of the means of ensuring the production of reliable and environmentally friendly material in construction and manufacturing industries is the reuse of industrial by-products or waste (Gola et al., 2015; Hospodarova et al., 2018), as well as the use from a renewable source. Besides, the potential application of wastepaper fibre in cementitious and earth-based material has economic and environmental benefits since the cost of recycled wastepaper fibre compared to a virgin pulp is only of the order of 15-25% (Coutts, 1989). Furthermore, several methods have been used to obtain pulp from various sources: alkaline-based (Kraft and soda pulping) (Jansson & Brännvall, 2014); acid-based (Lam et al., 2004) and solvent-based (organosolv pulping) (Correia et al., 2015; Ruzene et al., 2007).

This chapter assesses the response to pulping and bleaching of renewable and sustainable biomass from different tropical plants sources in order to evaluate their potential as a single or blended feedstock for nanocellulose production. The recycling process of municipal waste fibre such as waste carton boxes was also studied to promote the conservation of plant resources, as well as the circular economy. This chapter presents the preparation and physical-chemical/ morphological characterization of pulp and nanofibrillated cellulose. A comparative evaluation on the production yield, crystallinity index, and morphological properties of pulp based on the fibre type and the pulping technique was carried out in order to assess their potential application as reinforcement in an inorganic matrix.

3.2.Materials and Methods

3.2.1. Materials

The raffia fibre used in this work was extracted from the stem of *Raphia vinifera* originating from Melong, Cameroon. It was soaked in distilled water at room temperature for 2 days (Tagne et al., 2018). The ambarella was extracted from *Spondias dulcis* piths after the separation from ripe fruit purchased from a market in Douala, Cameroon. Those piths were soaked for 5 days in water and washed to separate the nut from the fruit. The cassava bagasse was obtained from a cassava paste ("water-fufu") production at Lelem, Cameroon and washed with water to remove excess starch. Bamboo pulp was produced from *Bambusa vulgaris* chip harvested at the University of Sao Paulo, Pirassununga, Brazil, while the waste carton boxes were collected in Pirassununga supermarket bins.

3.2.2. Methods

3.2.2.1. Chemical Composition

The wax content was obtained by boiling the crushed feedstock in a benzene/ethanol mixture (2:1 volume/volume) for 10 h (Shamsul Alam & Khan, 2007). The collected sample was oven-dried at 70 ^{o}C for 24 h to determine the amount of fatty and waxy content by measuring the loss in weight of samples. The determination of the lignin content was carried out by the Klason method, the crushed and extracted fibres with dichloromethane before being hydrolyzed in a 72 % solution of sulphuric acid (Sreenivasan et al., 2011). Hemicellulose content in the samples was determined using the Neutral detergent fibre method (AOAC, 1997).

3.2.2.2. Soda and organosolv pulping and acid-chlorine bleaching

Soda pulping was performed according to (Gonzalez et al., 2008). The dried specimens were manually chopped to a length size of about 1 cm to produce the feedstock. The specimen was introduced inside a solution of 6 % w/w of NaOH at 160 ^{o}C and maintained for 90 min. The organoslov pulping was performed by introducing 200 g of each feedstock into a pressurised stirred 4848 reactor (Parr Instrument Company, USA), with temperature controller and capacity of 7 L. The raw materials were immersed in an ethanol/water solution (1:1) at a solid/solvent ratio of 1:10 (w/v) (Correia et al., 2015). The mixture was cooked at 190 ^{o}C for 2 h. The treated soda and organosolv pulps were transferred to a defibrillator (Marconi, model MA 758, Brazil), where 500 mL of 1 % NaOH was added to treat fibres for 3 min. Then, the mixture was filtered and washed with distilled water until neutral pH before the bleaching process. 200 g of the carton boxes material (manually cut at small pieces) was immersed in tap water for 48 h before transferring that to the defibrillator (Marconi, model MA 758, Brazil) to disintegrate for 1 h using about 10 L of water. Excess water from the mixture was drained manually using 0.044 mm sieve.

Dried pulp (10 g) was suspended in 333 mL of water and heated to a temperature of about (70 ± 5) ^{o}C. Sodium chloride (8.4 g) and glacial acetic acid (3.4 mL) were added and the temperature was maintained at (70 ± 5) ^{o}C for 60 min. Then the suspension was cooled in an ice bath to 10 ^{o}C for 2 h before filtered and washed with distilled water until a neutral pH.

3.2.2.3.Grinding Nanofibrillation of Cellulose

The production of nanofibrillated cellulose (NFC) was carried out with Supermasscolloider Mini, model MKCA 6-2 (Musuko Sangyo Co., Ltd, Japan). The solution of 2 % (w/w) consistency of pulps was introduced 10 times through the grinding system with rotation disk speed at 1700 rpm (da Costa Correia et al., 2016).

3.2.2.4.The Yield of Pulping Processes

The evaluation of the yield from the pulping processes was estimated by equation (1).

$$\%R = \frac{m_p}{m_f} \times 100 \qquad (1)$$

Where % R is processed yield, m_f is the dry mass (g) of natural fibre and m_p is the dry mass of the pulp (g).

3.2.2.5.Structure Evaluation of Natural Fibres, Pulps and Nanofibrillated Cellulose

The crystallinity of raw material, pulp and NFC was examined using a Rigaku Miniflex 600 diffractometer, model RU 200B (Rigaku Corporation, Japan) and the procedure was explained elsewhere (Correia et al., 2015). The evaluation of the crystallinity index (CrI) was carried out by using the empirical method (Segal et al., 1959) according to Equation (2).

$$\text{CrI}=100 \times \frac{(I_{002}-I_{am})}{I_{002}} \qquad (2)$$

Where, I_{002} is the value of the intensity of (002) reflections corresponding to 22° -23° (2 θ), and I_{am}, diffraction intensity corresponding to 18° -19° (2 θ). The values were obtained directly from the XRD diffractogram of raw materials, pulp and nanofibrillated cellulose (Buschle-Diller & Zeronian, 1992).

3.2.2.6.Fourier Transform Infrared Spectroscopy

FTIR was used to determine the alterations in both the functional chemical groups and cellulose structure after the pulping process. The spectra were obtained by a Thermo Scientific NICOLET iS5 (Thermo Fisher Scientific Company, USA), used as KBr pellet, using a spectral range of 400–4000 cm^{-1}. Spectral absorbance was recorded as a function of wavenumber.

*3.2.2.7.*Morphological Characterization of Pulps and *Nanofibrillated Fibres*

Morphological properties (e.g., length, diameter, curls, and kinks) of bleached and unbleached pulp were analyzed by a PulptecTM MFA-500 Morphology Fibre and Shive Analyser – MorFiTrac, (University of Sao Paulo, Brazil). Cellulose pulp image (Figure 7b, c) was collected by a ZEISS microscope Axio Imager.A2m, equipped with an AxioCam MRc digital camera (Carl Zeiss Microscopy, USA).

The morphology of nanofibrillated cellulose was evaluated with Atomic Force Microscopy (AFM) (NT-MDT Spectrum Instruments, USA). All images were obtained in tapping mode with a scan rate of 1 Hz and using Si tips with a curvature radius of 14 nm and angle of sloping tip wall of about 12°. A drop of diluted nanofibres aqueous suspension (sonicated) was allowed to dry on an optical glass substrate at room temperature and analyzed subsequently. The width of the nanofibre was estimated with Image J software (NIH, USA) from the average of 22 fibres per image.

The morphology of raffia fibre was observed under a ZEISS EVO/ LS10 Scanning Electron Microscope (SEM), ZEISS EVO/ LS10 Scanning Electron Microscope (SEM), (Carl Zeiss Microscopy, USA). The raffia fibre was cut by razor blade before depositing in aluminium tape to observe the cross-section.

Transmission Electron Microscope (TEM) images were taken using an FEI Tecnai F30 at 80 kV acceleration, (FEI Company, USA). The samples were prepared according to (da Costa Correia et al., 2016). The width of the nanofibrillated cellulose was calculated from the TEM images using software Image J. An average of 32 fibres was measured.

3.3.Results and Discussion

3.3.1. Chemical composition

The average chemical compositions of *Raphia vinifera*, *Spondias dulcis* pith and *Manihot esculenta* are presented in Table 3.1.

Table 3. 1 Chemical composition of raffia fibre, ambarella and cassava bagasse

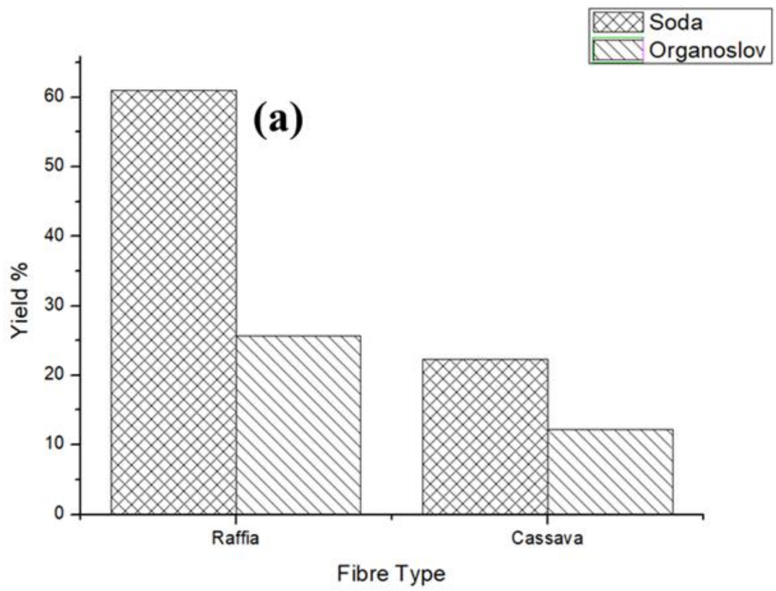
Sample	**Lignin (%)**	**Cellulose (%)**	**Hemicellulose (%)**	**Fatty and waxy mater (%)**	**Ash content (%)**	**Aqueous extract (%)**
Raffia fibre	3.66	48.32	24.90	1.34	1.04	2.59

Ambarella	19.86	12.37	22.40	18.87	16.05	2.62
Cassava bagasse	2.08	8.66	5.59	1.00	0.84	0.92

The cellulose content of raffia fibre is within the average found by organosolv pulping of non-wood and wood sources (Jones et al., 2017) indicating that raffia fibre would be a more attractive source of micro/nanofibres. The cellulose content for cassava bagasse agrees with values reported in the literature (Leite et al., 2017), on a study carried out on the isolation and characterization of cellulose nanofibres from cassava root bagasse and peelings. Furthermore, the wax and ash content of raffia and cassava fibres are within the range of the ash content of biomass used for pulp production (Jones et al., 2017). On the other hand, ambarella had a higher percentage of lignin (19.9 %), fatty and waxy matter (18.9 %) and ash content (16.1 %) relatively to the other fibres indicating that use of ambarella for production of pulp will not have a good economic value because the high amount of lignin will need more time, chemical and energy to have a cellulosic pulp.

3.3.2. The yield of Pulp Produced by soda and organosolv pulping

Figure 3.1 presents yield after pulping of raffia and cassava bagasse. The results of raffia soda pulping are consistent with other authors (Correia et al., 2015; El Omari et al., 2017; Hosseini et al., 2017). In both fibres, soda pulping gives a higher yield compared to the organosolv pulping process. This may be due to higher residual lignin in soda pulping and partial removal of non-crystalline cellulose during organosolv pulping. The organosolv pulping method appears more effective on delignification than soda, but the yields are lower, suggesting the loss of cellulosic materials during organosolv pulping. From Figure 3.1b, it is clear that the production of cellulose pulp from raffia fibre and cassava bagasse exhibits significantly low efficiency compared to bamboo fibres and waste carton boxes. The yield of bamboo (55.6%) is consistent with the value found in the literature, for example Correia et al., (2015) obtained 51.0 % from organosolv pulping of *Bambusa tuldoides* and Ciaramello (1970) obtained respectively 50.3 % and 55.0 % using soda and sulphate pulping of *Bambusa tuldoides*. The explanation of this low production efficiency from cassava plant may be due to a small amount of cellulose compound present in the cassava bagasse (Table 3.1).



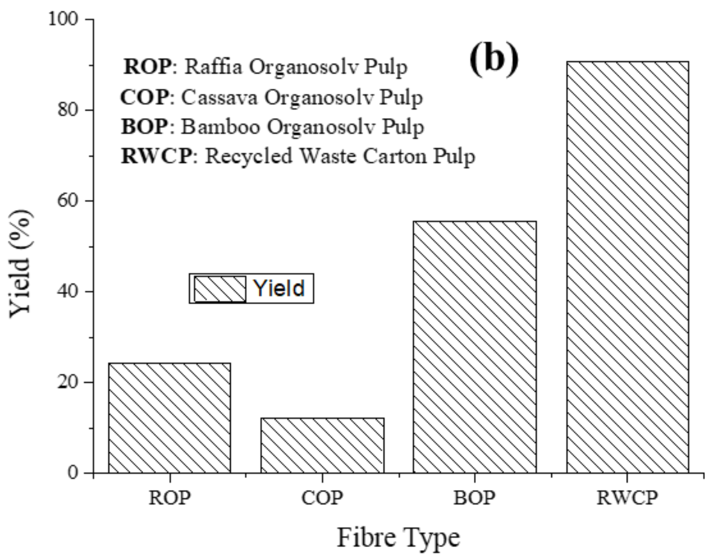


Figure 3. 1 The yield of pulping production of (a) raffia fibre and cassava bagasse from soda and organosolv process and (b) virgin resources (raffia, cassava and bamboo fibre) and waste

Previous studies using organosolv pulping, have demonstrated relatively high yields values of 54.5 % for sugarcane bagasse (Ruzene & Gonçalves, 2003), 56.8 % for sisal fibre (Joaquim et al., 2009) and 51.0 % for bamboo chips (Correia et al., 2015). However, the yield of cassava bagasse was 22.4 % with soda process and 12.3 % for the organic solvent process. This low yields could be attributed to the low amount of cellulose content of the raw material (Correia et al., 2015). Other studies reported similar yield (10.5-12.3%) for the extraction of cellulose from cassava root bagasse and peelings by acid hydrolysis method (Leite et al., 2017). Furthermore, previous studies on soda pulping process on oil palm frond fibre gave a yield of 32-44 % (Rosli et al., 2004). Oil palm frond fibre and raffia fibre are palm species native to Africa. However, the recycled waste carton pulp presents a higher production yield of 90.8 %wt. compared to bamboo organosolv pulp, which suggests high efficiency of the recycling process.

3.3.3. Physical-chemical Characterization of Raw Materials and Pulps

The crystallinity index, CrI of raw material and pulp is calculated based on X-ray patterns of Figures 3.2, 3.3, and 3.4. Generally, it shows that the pulping and nanofibrillation increases the crystallinity index of the raw materials. Diffractogram of cassava bagasse in Figure 3.3 shows two peaks at $2\theta = 17.0^{o}$ and $2\theta = 22.8^{o}$ while cassava pulp produced using soda pulping shows mean peak at $2\theta = 16.3^{o}$ and $2\theta = 22.1^{o}$. Cassava bagasse and cassava soda pulp have a typical crystal lattice for cellulose I, which the intensity of the main peak changes in the intensity of diffraction peaks indicating a modification in the structure or crystallinity cellulose molecular chain regularly (Adel et al., 2011). The high crystallinity of raffia soda pulp and bamboo organosolv pulp indicates higher perfection of the crystal lattice due to pulping. The crystallinity index in raffia fibre and pulps produced were found to be 45.9 % and 51.1 % respectively, while that for cassava bagasse fibre and pulps produced were found to be 9.7 % and 38.5 % respectively. Bamboo fibre provides highest crystallinity index, evaluated respectively at 61.2% and 71.2 for fibre and pulp. Other studies on cassava bagasse also reported low crystallinity index before and after fibrillation (around 14.52 % for cassava bagasse and 39.37 % for cassava nanofibre) (Wicaksono et al., 2013). The increase in the overall order of the hydrolysed fibres can be attributed to the removal of the hemicelluloses and lignin which exist in amorphous regions during the chemical treatment (Rosazley et al., 2016). Furthermore, Figure 3.3 show that after pulping of cassava bagasse, there is remarkable disappearance of peaks between 15.0^o and 17.0^o. This may be attributed to the removal of starch, lignin and hemicellulose on the cassava structure. The results of Figure 3.5 demonstrate that chemo-mechanical treatment increases the degree of crystallinity of raw raffia fibre, cassava bagasse, and bamboo fibre. The results also revealed that there is a large difference in crystallinity between the raffia fibre (46 %) and the raffia nanofibrillated cellulose (62 %). It can be seen that nanofibre is more crystalline than cellulose pulp and feedstock. Similar results have been reported elsewhere (Wicaksono et al., 2013). The organosolv pulping process of bamboo and the recycling process waste carton was effective since the value of yield (Figure 3.1 b) and crystallinity index (Figure 3.5) were within the average found in the reaction carried out by the conventional method.

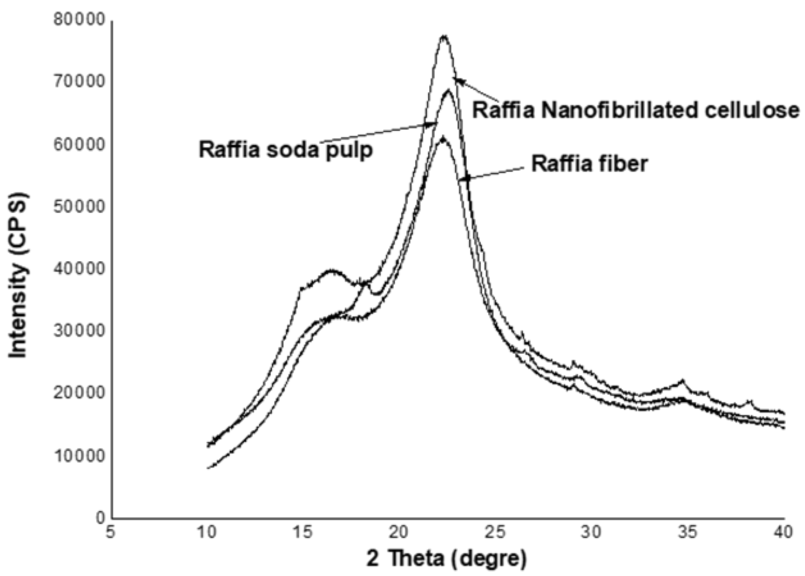


Figure 3. 2 X-ray diffraction pattern of raffia fibre and soda pulp

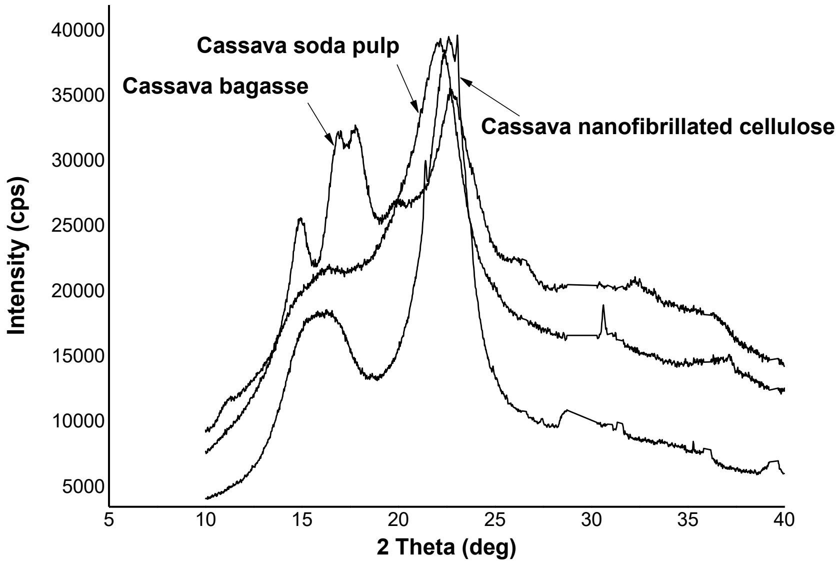


Figure 3. 3 X-ray diffraction pattern of cassava bagasse and soda pulp

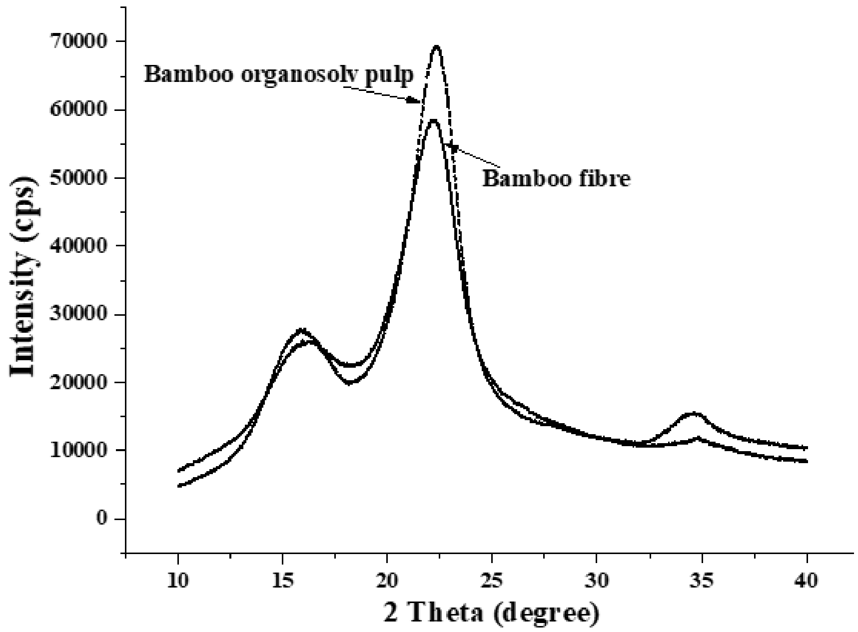


Figure 3. 4 X-ray diffraction pattern of bamboo fibre and organosolv pulp

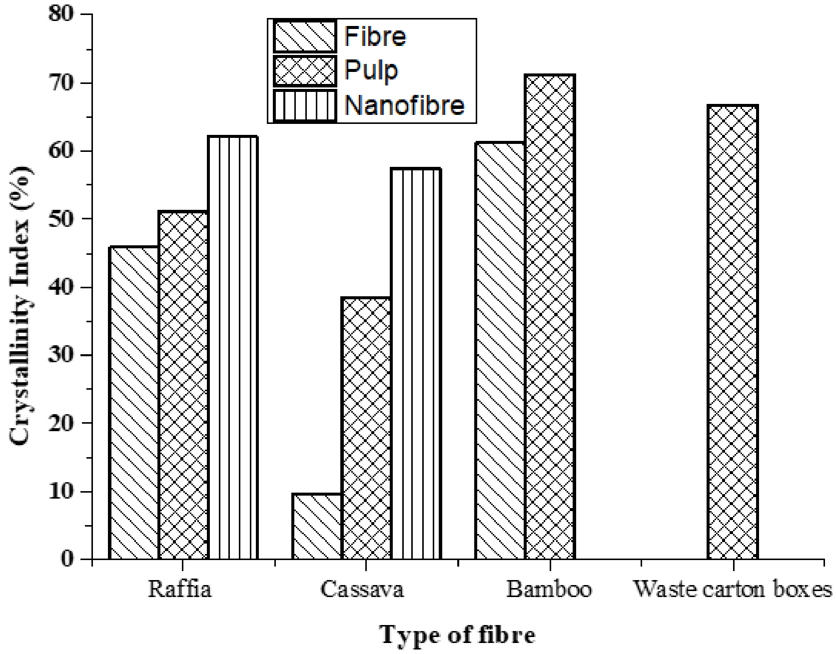


Figure 3. 5 Crystallinity index of cassava and raffia, bamboo and recycled waste carton before and after the pulping process

FTIR spectra presented in Figures 3.6 and 3.7 show the changes in the chemical structure of cellulose after pulping and bleaching of raffia fibre and cassava bagasse. For the non-equivalent C-O-C bond, in the region of band intensity of 900 cm^{-1}, which is related to the amount of crystalline matter in the cellulose structure. However, this peak disappeared completely in the spectra of bleached and unbleached cassava and raffia pulp, which can be attributed to the removal of most of the hemicellulose after the pulping process (Alemdar & Sain, 2008a). The peak at wave number 1242 cm^{-1} in the raw raffia fibre and cassava bagasse was associated to the C-O stretching of the aryl group in lignin. The disappearance of this peak in the unbleached and bleached pulp was believed to be due to the removal of lignin (Jonoobi et al., 2009). The presence of a remarkable peak at 1748 cm^{-1} in the spectra of untreated raffia fibre is associated with the C=O stretching of the acetyl group in hemicelluloses (Jonoobi et al., 2011). The peak may also be attributed to-either the acetyl and uronic ester groups of the hemicellulose or the ester linkage of the carboxylic group of the ferulic and p-coumaric acids of lignin and/or hemicelluloses (Alemdar & Sain, 2008a; Li et al., 2009). The peak at 1640 cm^{-1} on all the spectrum is attributed to the vibration mode of the cellulose water Correia et al., (2016; Li et al., 2009; Sun et al., 2005). Although the double bond between carbon and carbon C=C at the band of 1512 cm^{-1} represents by the peak in Figure 3.6 and 3.7 may be from the water, it was attributed to the aromatic ring in the lignin since this peak disappeared in other spectra due to the removal of lignin (Li et al., 2009). Therefore, it should be highlighted that this peak disappeared completely from the spectra of the treated fibres because of the removal of lignin and most of the hemicellulose during the chemical and mechanical treatments. The pulping process caused the modification in the cellulose structure, which made it less hydrophilic due to the exchange of the hydroxyl groups by the double bonds. No significant difference was found between the spectra of unbleached and bleached cellulose pulp, suggesting that the molecular structure of cellulose was not changed during bleaching. The crystallinity index of the pulp and nanofibrillated cellulose increase and the disappearance of those specific peaks on the infrared of pulp confirms the removing of the lignin, some carbohydrate and impurity during pulping.

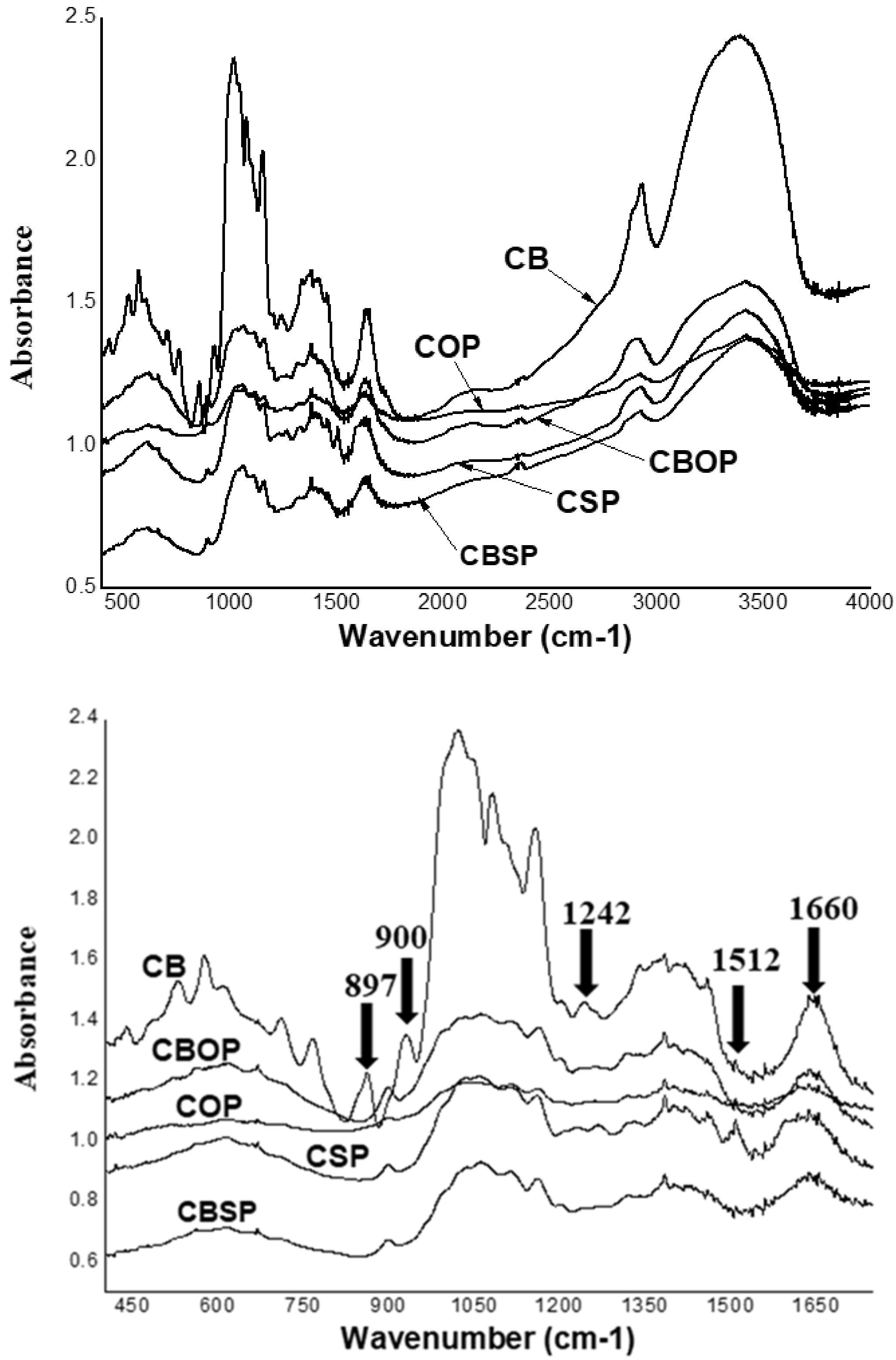


Figure 3. 6 Infrared of cassava bagasse (CB), cassava soda pulp (CSP), cassava bleached soda pulp (CBSP), cassava organosolv pulp (COP) and cassava bleached organosolv pulp (CBOP) (a) total view and (b) detail view

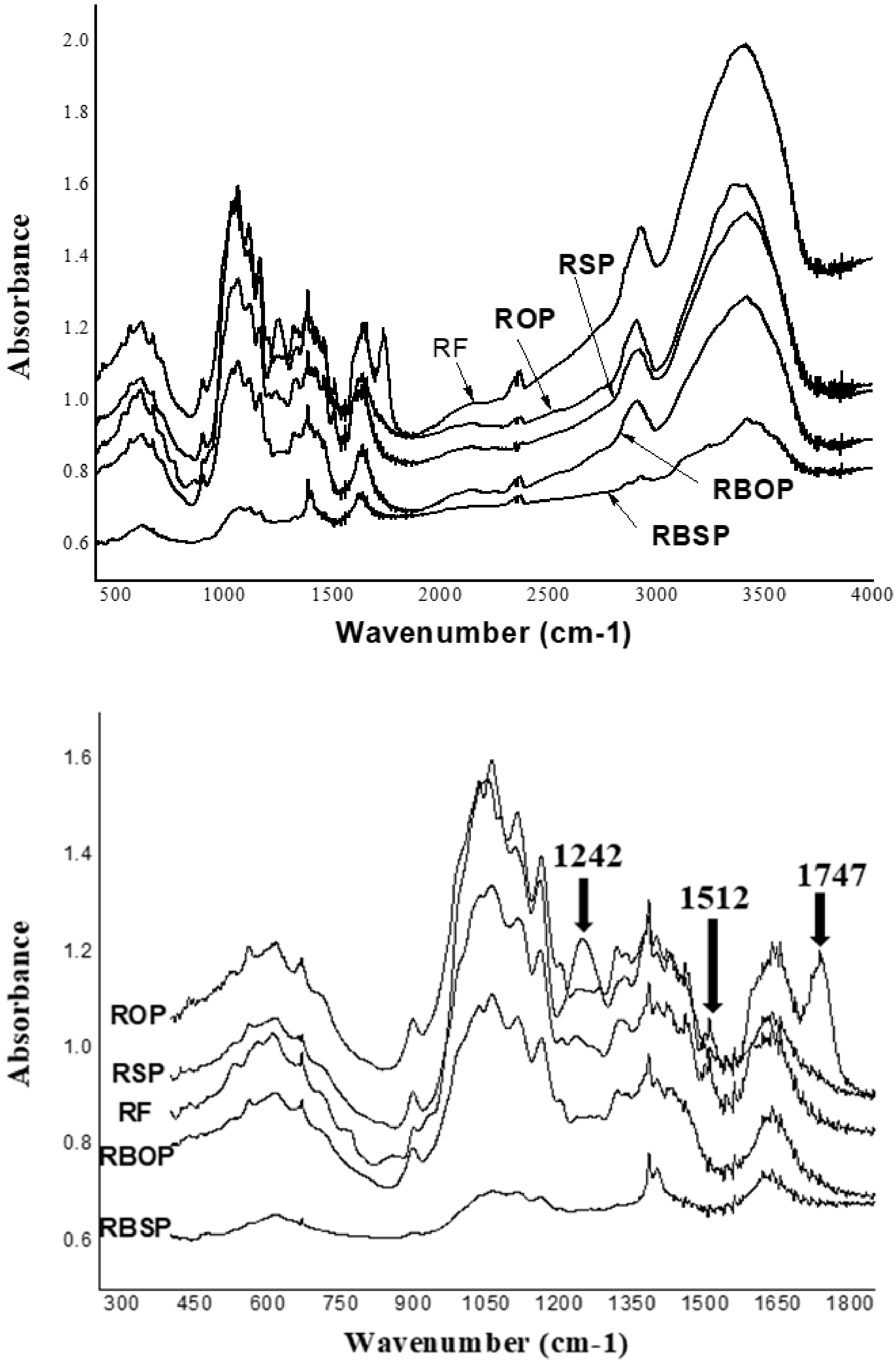


Figure 3. 7 FTIR spectra of raffia fibre (RF), raffia soda pulp (RSP), raffia bleached soda pulp (RBSP), raffia organosolv pulp (ROP) and raffia bleached organosolv pulp (RBOP) (a) global view and (b) detail view

3.3.4. Morphological characterization of bleached and unbleached pulps

The results of the morphological characterization of pulps including curls and kinks (corresponding to defects and distortions that occur during the pulping process of fibres and affect the resistance and break energy of the pulp) obtained for raffia fibre and cassava bagasse are shown in Table 3.2.

Table 3. 2 Morphological characteristics of raffia, cassava, bamboo and recycled waste carton pulp

	Average length (μm)	Average width (μm)	Aspect ratio	Coarse-ness (mg/m)	Fine content millions/g	Kinked fibre content (%)	Curl index (%)	fibers content (10^6 /g of pulp)
Soda raffia unbleached	1135 ± 43	30.80 ±1.20	36.86	0.27	26.10	21.41	9.89	5.50
Soda raffia bleached	1093 ± 28	29.30 ±0.80	37.30	0.03	17.19	28.26	11.59	47.86
Soda cassava unbleached	215 ± 32	32.90 ±1.30	6.53	0.19	10.31	17.45	8.03	1.21
Soda cassava bleached	372 ± 17	31.70 ±0.80	11.74	0.09	12.63	24.84	7.48	30.32
Organosolv raffia unbleached	371 ± 17	31.40 ±0.75	11.82	0.13	10.56	28.99	8.35	71.71

Organosolv raffia bleached	379 ± 20	31.90 ±0.76	11.88	0.09	9.45	26.79	8.04	46.90
Organosolv cassava unbleached	551 ± 17	24.50 ±0.84	22.49	0.05	6.19	14.80	7.64	22.02
Organosolv cassava bleached	816 ± 17	24.00 ±0.84	34.00	0.06	6.87	18.99	8.01	32.72
Organosolv bamboo	995± 513.4	16.00 ±12.98	58.73	-	-	-	-	-
Recycled waste carton	601.4± 342.7	12.81 ±7.11	46.74	-	-	-	-	-

The average number of fibres per gram represents the density of raffia fibre and the density increases with the bleaching process. The percentages of curl (bending) and the kink (twisted) levels of the fibres presented a slight tendency to increase during the bleaching process. A similar feature was shown by (Page et al., 1985). In the papermaking industry, the increase of the curl index has a significant impact on the mechanical properties of the paper structure such as the reduction of the tensile strength (Małachowska et al., 2019). The aspect ratio is an important parameter which affects significantly the amount of stress dissipated from the matrix to the embedded fibres when fibres are used as reinforcement in composites (Claramunt et al., 2011). The coarseness of the fibre is defined as the amount of mass in milligram per unit fibre length (Mármol et al., 2017). Coarseness is used as an indirect indicator of the fibres strength and deformability and that indirectly characterizes the thickness of the fibres (Ramezani & Nazhad, 2004). The morphological properties (length, width, coarseness and curl) of raffia soda pulp are similar to eucalyptus pulp (Małachowska et al., 2019) and hemp talks, birch and pine pulp (Danielewicz et al., 2019). The results show that the pulps produced with soda method were more affected by chemo-mechanical process than those produced with the organosolv method. This was confirmed by the high value of the deformation of fibre (kink and curl) during soda pulping. From Table 3.2, it can be seen that the bleaching process increases the value of the aspect ratio. In addition, the higher value of the aspect ratio is obtained with organosolv bamboo pulp (58.73) and recycled waste carton pulp (46.74) (Figure 3.8a and 3.8b), which indicated the potential application of bamboo and recycled pulp as a reinforcing element in cementious and earth-based matrix. The mean width and length of raffia bleached soda pulps were 29.3µm and 1093.0 µm respectively and the aspect ratio was 37.30. In the literature, autoclaved bamboo Kraft pulp has a mean length 1.7mm and width 20.0 µm (Robert S P Coutts, 2005) and bamboo organosolv pulp has length 2.7mm and width 76 µm and aspect ratio of 40.2 (Correia et al., 2015). The characteristics of raffia soda pulp and cassava organosolv pulp are similar to those mentioned in the literature (Coutts, 2005; Correia et al., 2016). Therefore, the pulps sourced from these materials can be considered as an alternative and useful in reinforcing the material.

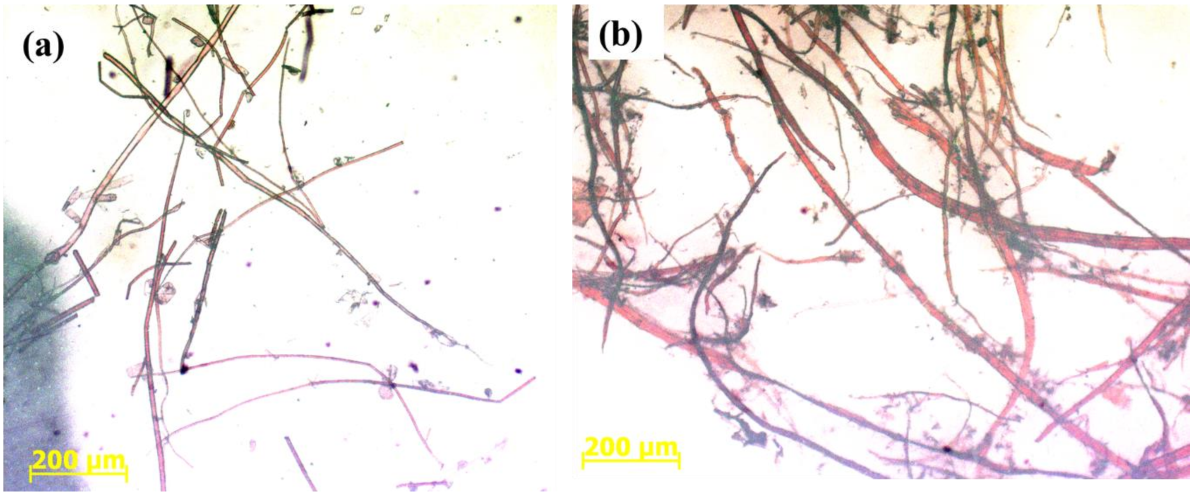


Figure 3. 8 (a) Optical image of bamboo organosolv pulp and (b) recycled waste carton pulp

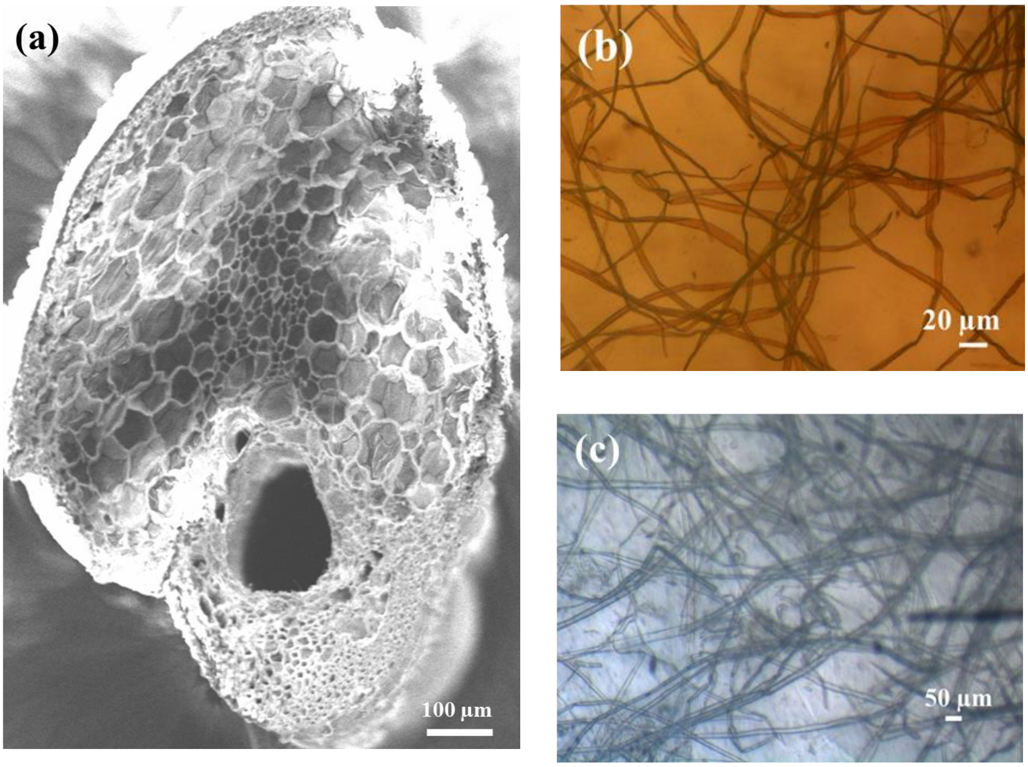


Figure 3. 9 (a) SEM image of cross-section raffia fibre and optical microscope image of (b) raffia soda pulp and (c) raffia organosolv pulp

The morphological characterization of nanofibrillated cellulose result obtained from Raffia fibre and cassava bagasse with Taping Mode Atomic Force Microscopy are shown in Figures 3.10-3.13 and widths are presented in Table 3.3. Figure 3.9 reveal the porous microstructure of raffia fibre and Figures 10-12, show the inhomogeneity of nanofibrillated cellulose and includes nanofibre bundles and nanofibrillated fibres. This may be due to the interaction and agglomeration of the hydroxyl groups on the surface of the fibres (Correia et al., 2016; Siró & Plackett, 2010).

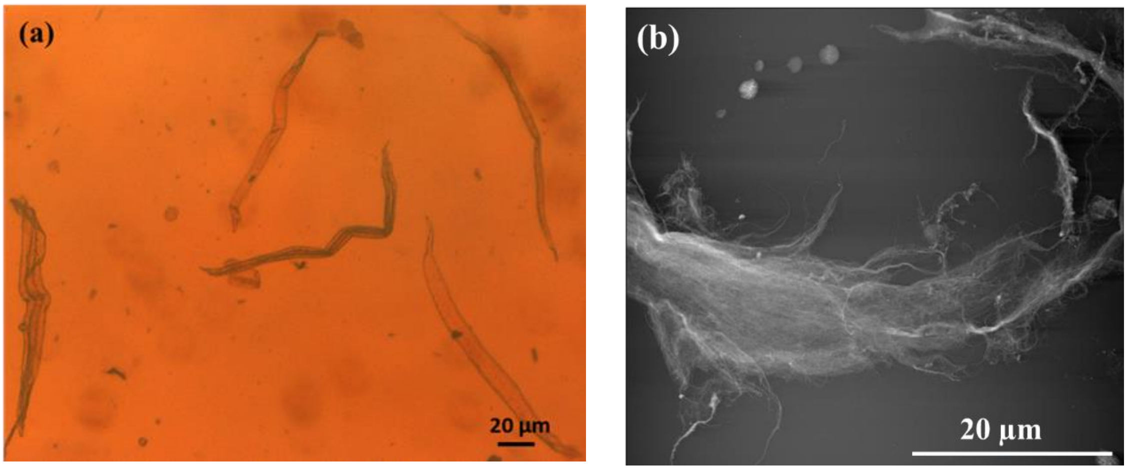


Figure 3. 10 (a) Optical image of cassava soda pulp and (b) nanofibrillated cellulose from AFM

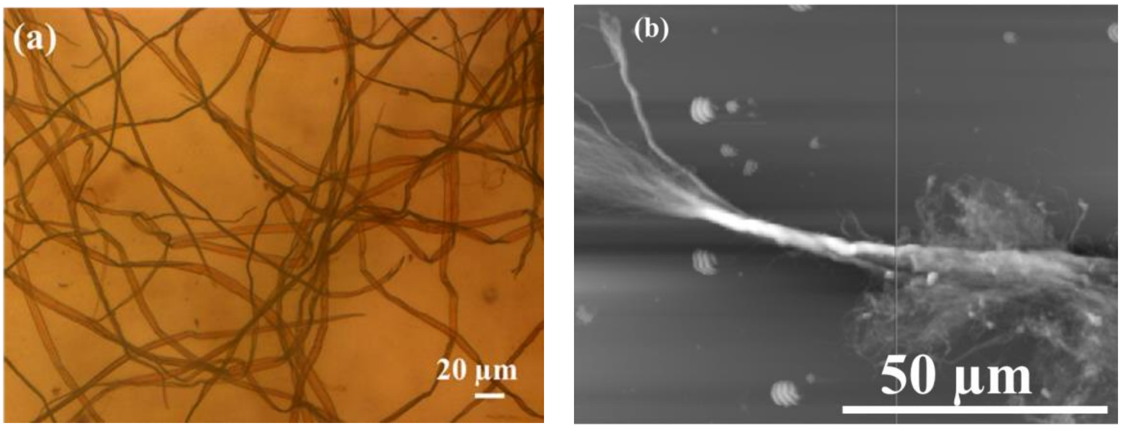


Figure 3. 11 (a) Optical image of raffia soda pulp and (b) nanofibrillated cellulose from AFM

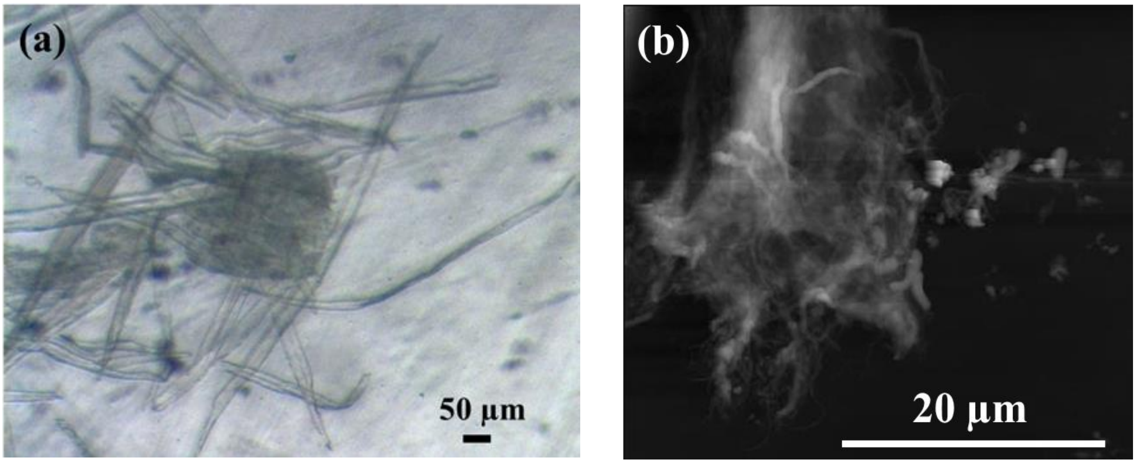


Figure 3. 12 (a) Optical image of cassava organosolv pulp and (b) nanofibrillated cellulose from AFM

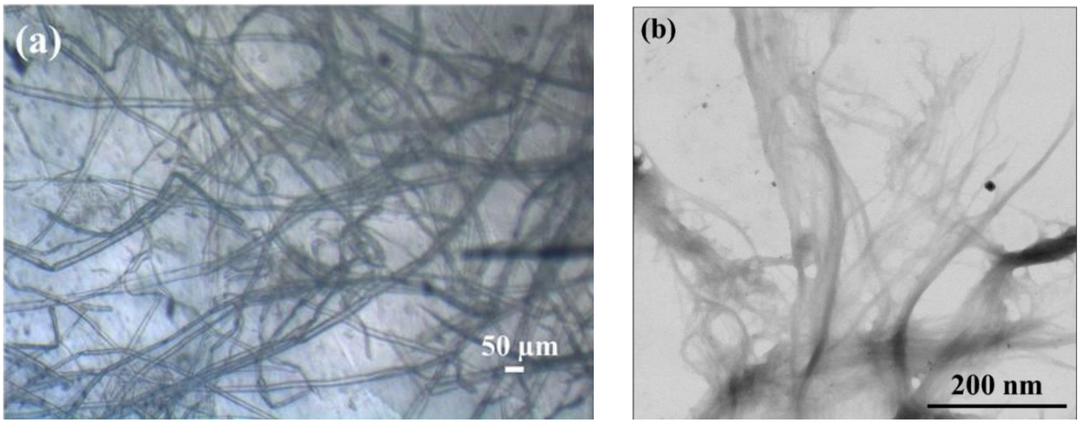


Figure 3. 13 (a) Optical images of raffia organosolv pulp and (b) nanofibrillated cellulose from TEM

The width of the fibre is evaluated with Image-J as presented in Table 3.3.

Table 3. 3 Width of nanofibrillated cellulose

Name nanofibrillated cellulose	**Average width (nm)**
Cassava soda nanofibrillated cellulose	154 ± 63
Raffia soda nanofibrillated cellulose	278 ± 67
Raffia organosolv nanofibrillated cellulose	110 ± 80
Cassava organosolv nanofibrillated cellulose	241 ± 60

The width of raffia and cassava nanofibrillated cellulose was estimated between 110 nm and 278 nm. Studies have been done on the isolation and characterization of nanofibres from agricultural residues (Alemdar & Sain, 2008b) with the results implying that the broad width distribution of wheat straw nanofibre and soy hull nanofibre obtained after mechanical treatment was respectively ranging from 10–80 nm and 20 to 120 nm. The high value of the width of raffia nanofibrillated cellulose may be explained by the initial high value of the real cross-sections (Figure 3.7), which is between 1.049 mm^2 to 1.190 mm^2 with equivalent width between 1.155 mm and 1.231 mm (Tagne et al., 2017).

3.3.5. Effect of pulping and bleaching process in the morphological properties of raffia and cassava pulp

According to the results of MorFiTrac equipment of Table 3.2, Figure 3.14 showed that raffia pulp produced using organosolv method had low aspect ratio, coarseness and high deformation (kink) compared to raffia fibre produced by soda method. This suggests that the raffia pulps produced via the organosolv method were more affected by the chemo-mechanical process than those produced using soda method. The aspect ratio is an important parameter which significantly affects the amount of stress dissipated from the matrix to the embedded fibres when used as reinforcement in composites (Claramunt et al., 2011). Despite the low yield of production, the cassava pulp presented a better aspect ratio after organosolv pulping and low deformation, hence it could be used for composite reinforcement application. The yield of production of cassava pulp was very low compared to the raffia fibre indicating that raffia fibre would be a more reliable source of pulp production than cassava bagasse and ambarella. Furthermore, the overall properties of Figure 3.14 reveal that raffia fibre has a better response to soda pulping, while cassava bagasse responded better to the organosolv pulping process. Thus, there is a feasibility of blended raffia soda pulp with cassava organosolv pulp to produce nanofibrillated cellulose.

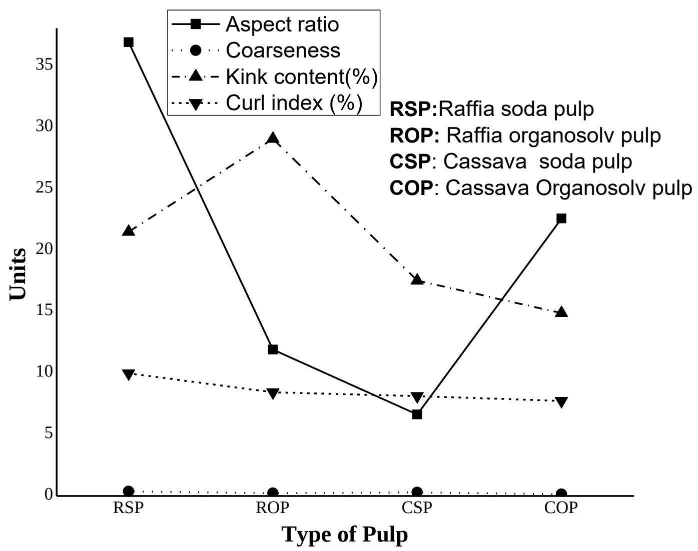


Figure 3. 14 Morphological parameters of raffia and cassava pulps

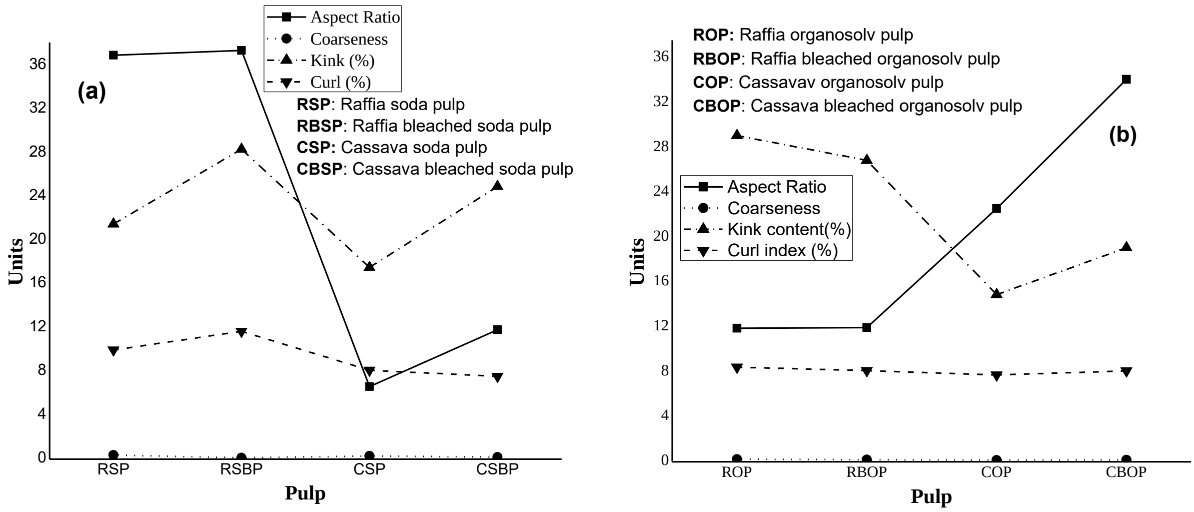


Figure 3. 15 Effect of bleaching process in the aspect ratio, coarseness, kink content and curl index of (a) soda process and (b) organosolv process

The results of Figure 3.15 reveal that the bleached process did not affect the value of the aspect ratio of raffia pulp and increase at 79.8 % and 51.2% respectively the aspect ratio of cassava soda pulp and organosolv pulp. Although, the coarseness of the pulp reducing during bleaching process due to the removing of the residual lignin and hemicellulose content of the pulp, the process increases the deformation (kink and curl) of the pulp fibre (Page et al., 1985).

3.3.6. Effect of aspect ratio, crystallinity index and production yield on the application cellulose pulp fibre

Figure 3. 16 gives the value of aspect ratio as well as crystallinity index and production yield of each type of cellulose pulp fibre. Organosolv process of bamboo and recycling process of waste carton provide cellulose pulp fibre with the highest aspect ratio, crystallinity index and production efficiency. Yoo et al. (Yoo et al., 2017) reported that steel fibre with a high aspect ratio provides a more effective improvement in the flexural strength, deflexion capacity and toughness of the cement composite compared to steel fibre with a low aspect ratio.

However, the production yield and crystallinity index bamboo and waste carton confirm the efficiency and selectivity of organosolv process of bamboo and recycling technique of waste carton, since the values are within the average found in reactions performed by conventional methods. Besides, the potential application of wastepaper fibre in cementitious, earth-based material has economic and environmental benefits since the cost of recycled wastepaper fibre compared to a virgin pulp is only of the order of 15-25% (Coutts, 1989). Their application as a reinforcement in building blocks may reduce pollution by greenhouse gas emission (Lelieveld & Crutzen, 1992) and promote the conservation of plant resources, as well as the circular economy.

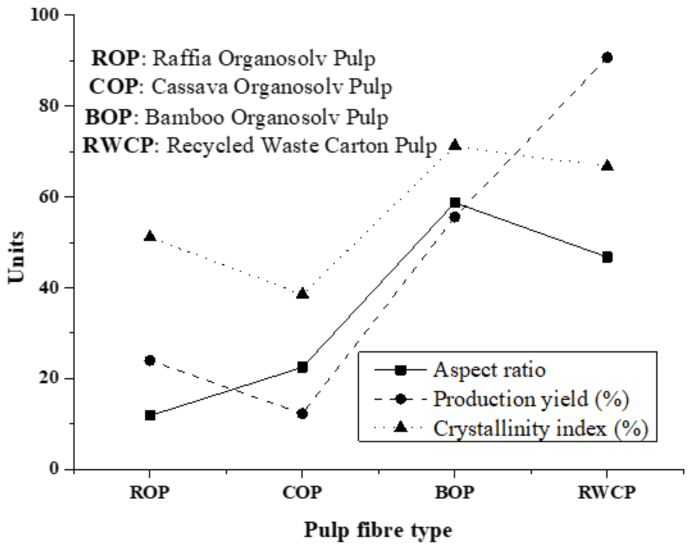


Figure 3. 16 Effect aspect ratio, crystallinity index, and production yield on the application of cellulose pulp fibre.

3.4.Conclusions

Pulp and nanofibrilated cellulose were produced from three tropical plants through organosolv and soda pulping methods. The effect of fibre type, method of pulping and bleaching was studied to evaluate the response of these fibres to pulping and nanofibrillation. The raffia fibre was found to have a very high cellulose content (48.32 %) compared to ambarella (12.37 %) and cassava bagasse (8.66 %) and showed a high potential for use as a source of cellulose pulp. However, the lower density and porous microstructure of raffia fibre greatly affect the production efficiency by the organosolv pulping process of raffia fibre. AFM analysis of the nanofibrillated cellulose show inhomogeneity of the fibres, with widths ranging between 110 nm and 278 nm for the raffia and cassava nanofibrillated cellulose. Also, the improvement in aspect ratio due to bleaching from 22.49 to 34.00 for cassava organosolv pulp and from 36.86 to 37.30 for raffia soda pulp, while the yield of the pulping process of raffia was practically more than double of the yield of cassava bagasse. Thus, there is the feasibility of blended raffia soda pulp with cassava organosolv pulp to produce nanofibrillated cellulose where these plants are prevalent. The organosolv pulping process has been adopted to produce cellulose pulp for use as reinforced in an earth-based matrix, as this process uses an organic solvent (e.g ethanol) which eliminates the need for recovery of inorganic reagents and eliminates the emission of sulfur, which offers economic and environmental advantages over the soda method. However, the result shows that organosolv pulp of raffia fibre and cassava bagasse have lower coarseness and defects (curl index for instance) compared to soda pulping. In addition, raffia pulp and cassava bagasse pulp obtained by an organosolv process has a low aspect ratio (Table 3.2) and production efficiency (less than 25 %wt.)(Figure 3.1) compared to other non-wood resources such as the bamboo (production efficiency higher than 50%wt.). Based on the satisfactory result of aspect ratio, production yield and crystallinity index, bamboo plant and waste carton boxes were adopted as a raw material for cellulose pulp for reinforcement in the earth-based composite. The reinforcement efficiency of bamboo cellulose pulp and recycled waste carton pulp on a stabilized extruded soil matrix is evaluated in the next section, in order to assess the possibility of replacing virgin fibre with waste from recycled fibre to promote resource conservation as well as circular economy and environmental impact.

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CHAPTER FOUR

4.0.Effect of cellulose pulp fibres on the physical, mechanical, and thermal performance of extruded earth-based materials[3]

4.1.Introduction

Environmental concerns associated with cement production and the growing interest in developing eco-friendly and low-cost houses solutions have led to the revitalization of earth-based materials as a sustainable resource (Maskell et al., 2014b; Ojo et al., 2019). However, earthen construction materials suffer from shrinkage cracking during moisture loss. Having brittle properties when exposed to load is another drawback of this material that leads to non-durability. The brittle nature of this class of material has been attributed to the electrostatic restrictions of ceramic ion-covalent bonds at room temperature (Correia et al., 2018). They thus require reinforcement to improve the resistance to cracking due to shrinkage and stress. Addressing these shortcomings may help to expand its application in the construction industry. Historically, the most common method used to minimize shrinkage cracking during the drying process and to improve flexural strength/ toughness has been the introduction of lignocellulosic fibres (Ojo et al., 2019). Advantages of lignocellulosic fibres compared to other reinforcing fibres are their availability, non-toxic character, low cost, and renewability (Soares et al., 2019). A wide range of natural fibre such as bamboo (Kolawole et al., 2017), sisal (Ojo et al., 2020), coconut husk (Danso et al., 2015), straw (Mustapha et al., 2016) has been used to reinforce earth-based blocks to improve their bending strength and fracture toughness. This improvement of the fracture toughness in soil composite was attributed to the crack bridging mechanism.

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However, only a few studies have examined the influence of the inclusion of cellulose pulp fibres on the drying shrinkage, thermal conductivity, and mechanical performance of earthen wall materials (Muñoz et al., 2020; Ojo et al., 2019).

According to Ahmad & Ibrahim (2010), drying shrinkage in a cementitious material is defined as the reduction in volume caused primarily by water loss during the drying process. This shrinkage on drying is due to the evaporation of water in the material and represents nearly 70% of the total shrinkage of the cemented material (Kodikara & Chakrabarti, 2001). Fundamentally, shrinkage cracking happens while the cement stabilized layer loses humidity to bordering materials or the atmosphere (Biswal et al., 2019). As a result, some studies have attempted to adopt different techniques, including the possibility of introducing natural and synthetic fibre to reduce shrinkage cracking in the cementitious and earth-based materials. For example, Ferrara et al. (2015) showed that the micro and nanoscale addition of cellulose-based materials create a porous network within the cemented matrix. Therefore, this addition contributes to reducing the autogenous and drying shrinkage of the composite. These phenomena are due to their hydrophilic character, water retention capacity and porous structure. Bouhicha et al. (2005) showed that the incorporation of straw in the soil material increases the flexural, shear, and compressive strength and reduces the drying shrinkage of the composite. This reduction of drying shrinkage is relatively linked to the shrinkage crack reduction of the composite. Due to the instability dimensional (swelling-shrink) of clay soil and cellulose fibre in the presence of moisture (Ghavami et al., 1999), analysis of drying shrinkage of cement stabilised clay soil materials reinforced cellulose pulp fibre should be required to expand its application in the construction industry.

Cellulose pulp is currently used as a reinforcement agent in the cementitious matrix for improving flexural strength and toughness. Correia et al. (2014) stated that the reinforcement of cement matrix with 6 and 8 %wt. of bamboo pulp is sufficient to provide the best properties before cracking and also to arrest crack propagation after an initial cracking of the composites. Due to their small diameter and hydrophilic surfaces, cellulose fibres fit well in the structure of the cementitious matrix. Therefore, they can provide a uniform and denser microstructure and consequently, contribute to a satisfactory balance of physical and mechanical performance of the composite (Peters et al., 2016). Moreover, cellulose pulp fibres in the cementitious matrix yield better fibre-matrix bond, reinforce efficiency and significantly increase the toughness and flexural strength compared with vegetable fibres in a strand form (Karade, 2010; Savastano et al., 2003). Nowadays, cellulose pulp reinforced cementitious matrix is widely used in the production of corrugated and flat sheets, roofing tiles, partition board (walls), and other materials for construction (Soares et al., 2019).

Cellulose pulp has shown significant improvement in the properties of cement-based matrices, however, a study on cellulose pulp fibres as reinforcement elements in the earth-based matrix is still limited. Recently, Ojo et al. (2019) used eucalyptus pulp fibre to strengthen alkaline activated clay material and (Muñoz et al., 2020) demonstrated that paper and pulp wastes fibre can improve the compressive strength and thermal insulation properties of adobe bricks. Thus there is a need to investigate the potential reinforcement of cement stabilized earth-based matrix to reduce the drying shrinkage and increase the flexural strength and toughness which can consequently expand their applications in construction materials. Ojo et al. (2019) investigated the impact of eucalyptus pulp on the physical and mechanical performances of alkali-activated clay materials. The results show that the addition of 0.5, 1 and 2 % of eucalyptus pulp volume ratio in the earthen composite provides no significant improvement to brittle failure in the flexural test. This outcome is in agreement with the finding of other studies reported the efficiency of cellulose pulp fibre reinforced cementitious matrix with the fibre content ranged from 6 to 10 %wt. (Correia et al., 2014; Joaquim et al., 2009; Tonoli et al., 2009). The authors concluded that 2 % of the volume ratio of eucalyptus pulp is not sufficient to provide a beneficial contribution to the earthen matrix. Therefore, there is a need to evaluate the reinforcement efficiency of earth-based composite reinforced with cellulose pulp fibre at the volume ratio greater than 2%. (Muñoz et al., 2020) studied the effect of paper and pulp waste on the compressive strength, thermal conductivity and toxicity of adobe bricks by varying the percentage of fibres up to 20%. The results reported an improvement in compressive strength of up to 190%, while thermal conductivity was reduced by up to 30% for 12.5% of the cellulose pulp content. In addition, the bending strength, toughness, pH value of the matrix environment and drying shrinkage of the earthen wall material reinforced with cellulose pulp fibres should be evaluated to promote and vulgarize the application of this material as building components and to predict the durability of the fibre.

Based on the World Bank report (Kaza et al., 2018), the estimation of the municipal solid waste generated in 2016 was 2,010 million tonnes and this is expected to enhance it to 3,400 million tonnes in 2050. In this report, paper and cardboard represented 17 % of global waste. Recycled waste paperboard fibre is a promising reinforcement agent, considering its global redundancy and nearly low cost compared to lignocellulosic sources. It might additionally contribute to resource conservation and waste reduction as any recycled waste. The biodegradation of wastes from vegetable resources in landfills, releases methane, a greenhouse gas that has a heating effect approximately 72 times that of CO_2 (Lelieveld & Crutzen, 1992). (Soroushian & Shah, 1993) reported that the recycled magazine paper fibres could replace up to 50% of the virgin fibres without significant damage to the character of fibre cement composites. Currently, the use of recycled wastes fibres became more attractive as reinforcement in the cement matrix the constructing roofs, partitions, and non-load bearing walls (Bentchikou et al., 2012). Another strategy to minimize the cost of earth-based building materials may be the use of waste/ recycled pulp as a reinforcement of earth-based walling materials.

Bamboo is a promising fibre source for pulping process and reinforcement in various composites as compared to other green sources as a result of its easy and fast growth, relatively low cost and worldwide abundance along with its high tensile strength and its importance as a great non-timber forest product and wood substitute (Correia et al., 2014).

Over the past decade, earth-based block material development has moved toward increasing thermal insulating property and reducing its weight. Utilising vegetal fibre in the soil matrix is sustainable, and resulting in a reduction of the weight and the thermal conductivity of the composite. (Laibi et al., 2018) showed that the inclusion of kenaf fibre at a ratio of 1.2 %wt. reduced up to 50 % of the thermal conductivity of earth-based material. (Binici et al., 2007) demonstrated that the addition of straw fibre in the mud bricks reduces the density, the thermal conductivity and contribute to preventing energy loss from the walling materials.

This investigation attempted to produce viable cellulose pulp-reinforced earth-based walling materials using non-conventional bamboo organosolv pulp and recycled waste carton pulp. The main objective was to study the reinforcement efficiency of bamboo cellulose pulp and recycled waste carton pulp on a stabilized extruded soil matrix, in order to assess the possibility of replacing virgin fibre with waste from recycled fibre to promote resource conservation. Especially, this work; (i) evaluates the effect of varying the fibre weight fraction on the modification of physical properties (drying shrinkage, apparent void volume, density, and water absorption) of cement stabilised soil; (ii) examine the change in flexural strength (dry and wet state) induced by fibre inclusion in the matrix; (iii) evaluate the optimum cellulose pulp content for efficient reinforcement capability and the possibility to replace virgin cellulose fibre with recycled waste carton pulp fibre for resources conservation and (iv) investigate the inclusion effect of cellulose pulp on the thermal conductivity of the soil composite.

4.2.Materials and methods

4.2.1. Pulp preparation and characterization

Bamboo pulp was produced from *Bambusa vulgaris* chip using organosolv method described by Stanislas et al. (2020). Recycled waste carton pulp was obtained from the following method. 200 g of the carton boxes material (manually cut at small pieces) was immersed in tap water for 48 h before transferring that to the defibrillator (Marconi, model MA 758, Brazil) to disintegrate for 1 h using about 10 L of water. Excess water from the mixture was drained manually using 0.044 mm sieve. Similar methods have been used to defibrillate raffia cellulose pulp (Stanislas et al., 2020).

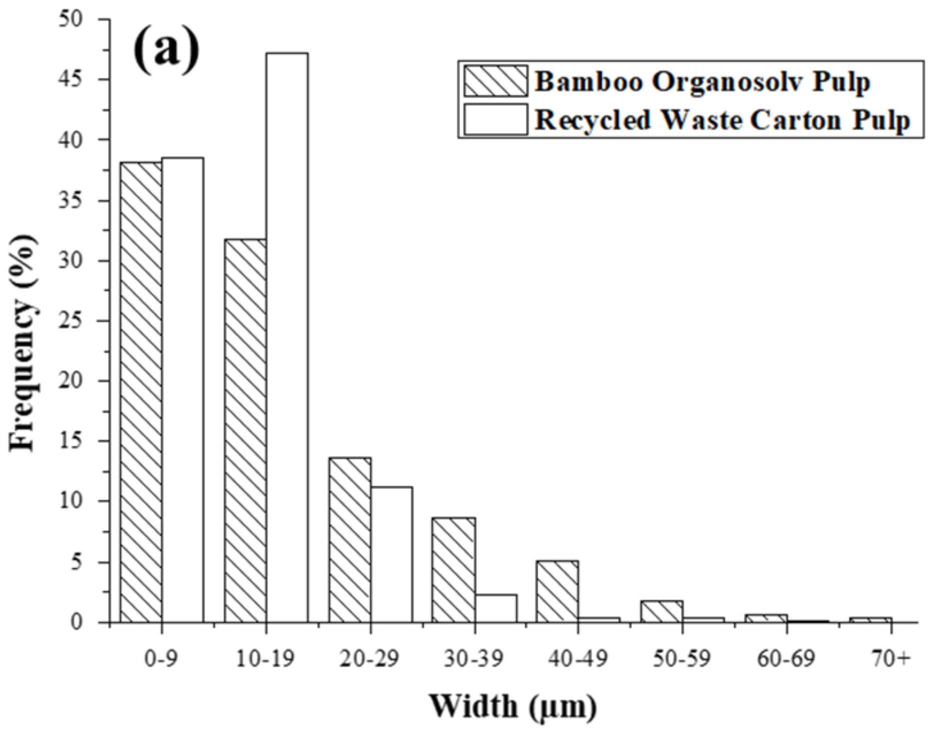
The specific density of bamboo organosolv pulp and recycled waste carton pulp was evaluated by the helium gas pycnometer (Quantachrome Instruments, USA). The morphological characterization of cellulose pulp was investigated with a ZEISS microscope Axio Imager.A2m, equipped with an AxioCam MRc digital camera (Carl Zeiss Microscopy, USA). Figure 4.1a and 4.1b present the distribution of width and length of cellulose pulp measured from about 500 fibres with Image J software. Table 4.1 presents the characteristic of bamboo and recycled waste carton pulp fibres.

Table 4. 1 Morphological and physical characteristics of cellulosic fibres

	Characteristic				
Type of fibre	**Width (µm)**	**Length (µm)**	**Aspect ratio**	**Specific density (g/cm^3)**	**The yield of production (%wt.)**

Bamboo organosolv pulp	16.94± 12.98	995.04 ± 513.38	58.73	1.558 ± 0.009	55.6
Recycled carton boxes pulp	12.81 ± 7.11	601.40 ± 342.74	46.74	1.566 ± 0.011	90.7

***Parameters are expressed as mean ± standard deviation**



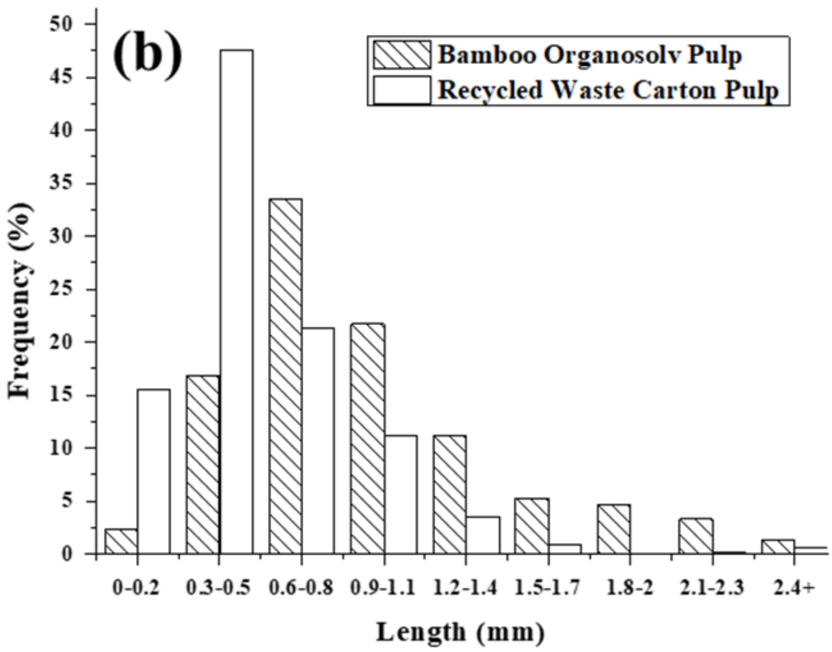


Figure 4. 1 Distribution of width (a) and length (b) of bamboo and recycled waste carton pulp

4.2.2. Characterization of the soil and cement

The soil used was donated by Top Telha Ceramic Tile, Brazil from their quarry located in Sao Paulo State. This soil is typically used for manufacturing commercial fired clay roof tiles. The obtained liquid limit and plastic limits were 42.5 % and 24.9 % respectively, based on the investigation conducted by Ojo et al. (2019). Table 4.2 contains the chemical composition and the size of the soil particle as reported by Ojo et al. (2019). According to the authors, and based on the unified soil classification system (USCS), the soil is classified as CL (inorganic clay of low plasticity). The cement used as a stabilizer was Ordinary Portland Cement, type CP V-ARI based on Brazilian Standards NBR 5733 and as per ASTM-C150 Type III standards, provided by Intercement Brazil. Figure 4.2 presents the particle size distribution of cement and clay soil using LA-950V2 Partica Particle Size Analyzer. It can be observed in Figure 4.2 that soil present a coarser distribution, with 75 % of particles < 50 µm.

Table 4. 2 Chemical composition, and class of particle size of the soil (Ojo et al., 2019)

Characteristics	**Chemical composition**				**Size of the particles**		
	SiO_2	Al_2O_3	Fe_2O_3	K_2O	Sand	Silt	Clay
Soil percentage (%)	70.39	13.31	4.09	4.97	35	33	32

The mineralogical confirmation of the starting soil samples was evaluated using X-ray Diffraction (XRD), model RU 200B, Rigaku Miniflex 600 diffractometer. The scan was performed from 10 to 70 θ (2 θ) at a rate of 2°/min and Cu-Kα at the laboratory of FZEA-USP, Brazil and the results revealed quartz as the main mineral on the sandy part of the soil. Trace of muscovite and microcline can be observed (Figure 4.3). The EDS result presented on Figure 4.4b is in agreement with XRF results (oxides content) of Table 4.2.

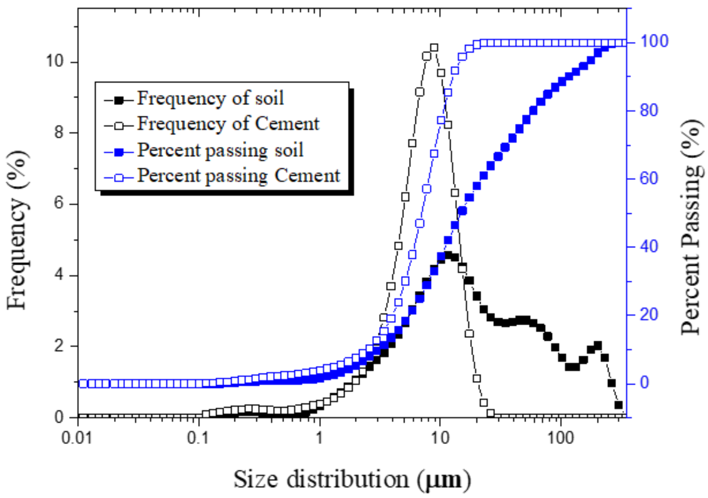


Figure 4. 2 Particle size distribution of soil and cement

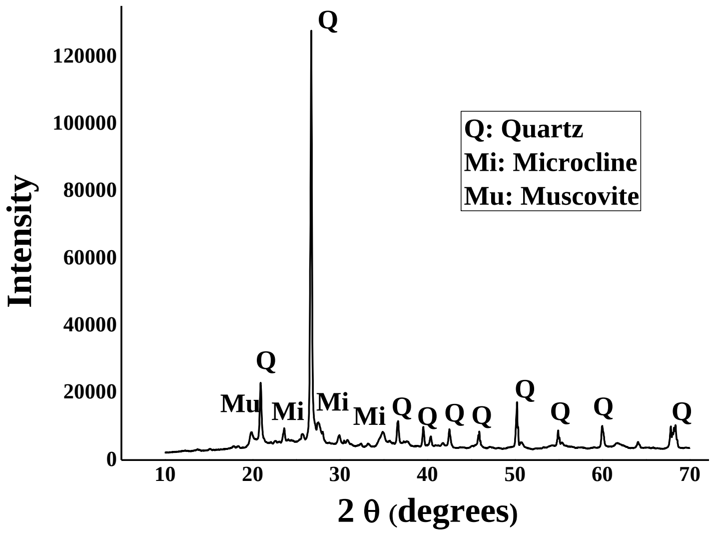


Figure 4. 3 XRD pattern of soil

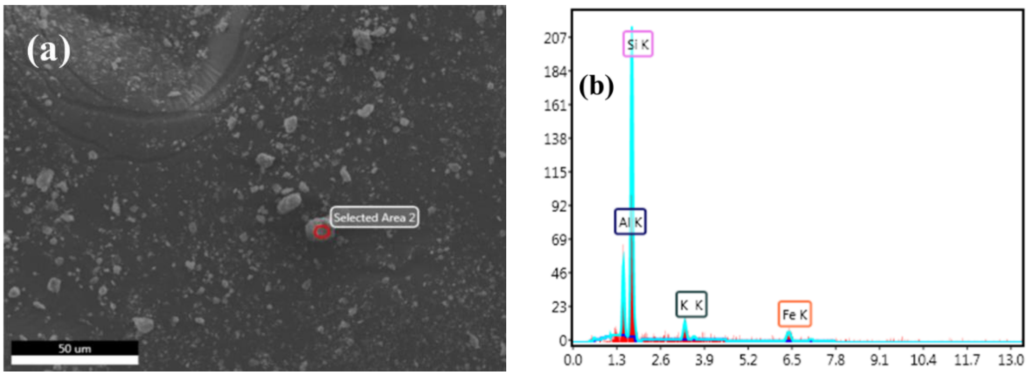


Figure 4. 4 (a) SEM image and EDS graph SEM image of soil

4.2.3. Specimen preparation, mix proportion and curing condition

4.2.3.1.Selection of cement percentage

For a preliminary investigation, there was a need to examine the influence of cement stabilization and curing condition on the development of the physical and mechanical performance of this clay soil which is currently used for fired roofing tile. Three different formulation (5, 7.5 and 10 %wt. of cement) and two curing conditions (24 °C, RH of 70 % and 60 °C, RH of 100 %) were applied. Cylindric samples with the diameter of 40 mm and the height of 80 mm were extruded using a Gelenski MVIG-05 laboratory extruder (Gelenski Ltda, Mandirituba, Paraná, Brazil) at the linear speed of about 4 mm/s. The specimens were prepared with approximate moisture content equivalent to soil plastic limit at 25 % (Maskell et al., 2012) to do the tests of compressive strength, water absorption, bulk density and apparent void volume. From Table 4.3, it can be seen that the water absorption and apparent void volume decrease with increasing bulk density and cement content in the clay soil, except the specimen containing 7.5 %wt. cement, curing at 60 ^{o}C. This outcome agreed with the findings reported by Reddy et al, (Venkatarama Reddy & Latha, 2014). The results show an overall improvement of the compressive strength with increasing cement content after 7 days. However, in the composite with 7.5 and 10 %wt. cement, the high temperature of curing accelerated the strength development, as compared to that cured at ambient temperature (24 ^{o}C) after 7-days. The compressive strength developed for all formulations and curing conditions exceeds the minimum compressive strength requirement of 3.2 MPa established for load-bearing concrete masonry building unit as described by IS 2185 (part 1) (BIS:2185, 2005). The composite with 10 %wt. cement content develops the highest strength in all the curing condition (Table 4.3). This improvement may be attributed to the production of a large quantity of binding cementitious products that are caused by hydration and pozzolanic reactions as previously reported by (Horpibulsuk, 2012). Considering a negative environmental impact due to the presence of stabilizer in the earthen building material, it is proven that stabilizing a thin earth wall present embodied energy or CO_2 advantages compared to using a thicker earth wall (Maskell et al., 2014a). The realization of a thin-walled building material must require a material with a higher compressive strength (Marsh et al., 2020). The formulation of 10 %wt. cement content and the curing condition of 60 °C and RH of 100 % were adopted as the processing condition for fibre reinforced composites. A similar value of the cement content has been reported by Maskell et al. (2014a), which calculate based on Global Warmish Potential and embodied energy the minimum amount of cement to stabilize extruded earth masonry. The authors reported that the maximum cement content to provide a minimum compressive strength of 3 MPa and accepted durability for the wall thickness of 100mm was 10.1 %wt. of dry soil. Horpibulsuk et al., (2010) studied the effect of cement (0-45 %wt.) on stabilized silty clay. The results showed that the increase in strength was significant at low cement content (0-10 %wt. cement) where cement per grain contact increases and, upon hardening, imparts a commensurate amount of bonding in the contact points. The specimen stabilized with 10 %wt. of cement develop the highest strength since early curing time. In addition, Malkanthi et al., (2020) studied earth block stabilized with various percentage of cement and lime. The result indicated that the cement stabilization achieved a wet compressive strength greater than that of the lime stabilization, and 10% cement stabilization achieved at 28-day wet compressive strength greater than 1.2 N/mm^2. Alavéz-Ramírez et al., (2012) evaluated the effect of soil blocks stabilized with 10 %wt. of cement, 10% of lime, and 10%wt. of lime combined with 10 %wt. sugar cane bagasse ash. The authors demonstrated that after 7, 14 and 28 curing days and under both wet and dry test conditions, specimen contained 10%wt. of cement showed low moisture content and high specific weight and compressive strength compared to blocks stabilized with lime and sugar cane bagasse ash.

Table 4. 3 Influence of cement content and curing condition on the physical and mechanical performance of earth-based composite after 7 days.

Curing condition	**Cement content**	**Water absorption (%w/w)**	**Bulk density (g/cm^3)**	**Apparent void volume (%v/v)**	**Compressive strength (MPa)**	**Specific energy (kJ/m^2)**
24°C, RH 70%	5%	28.8 ± 0.9a	1.47± 0.04a	42.4± 0.4ab	4.4 ± 0.7a	7.2 ± 1.3a
	7.5%	26.7± 1.0bc	1.53± 0.03ab	41.0± 0.8bc	5.4 ± 0.7a	9.7 ± 0.8b
	10%	26.1 ± 0.7c	1.56± 0.02b	40.5± 0.5c	6.6 ± 0.6b	9.6± 1.7ab
60°C, RH 100%	5%	29.1 ± 0.8a	1.48±0.02ab	42.9± 0.7a	4.8 ± 0.3a	8.0± 0.8ab
	7.5%	29.5 ± 0.3a	1.47± 0.01a	43.3± 0.3a	7.1 ± 0.4b	12.1± 1.0c
	10%	27.8± 0.6ab	1.50± 0.01ab	41.8± 0.6ab	9.6 ± 0.7c	13.5± 1.6c

*Value with distinct letters in the identic column mean statistical difference resulted from Tukey post-hoc test ($p < 0.05$)

4.2.3.2.Stabilized soil reinforced with cellulose pulp

Cement and dry clay soil were mixed as described in the previous section at high speed before the start of the fibres with further blending for 10 min. The proportion of 1 %wt. of cement content of water reducer polyether carboxylic (commercial name ADVA 190) was mixed to approximately 35 %wt. of water and used as rheological modifiers to improve pseudo-plastic performance of the composite (Santos et al., 2015). The paste was mixed for 5 min before it was transferred to the extruder. The cross-section of the utilized extruder was rectangular with a width/height ratio of 3.3 and it was running at a linear speed of around 4 mm/s. Samples of 160 mm×50 mm×15 mm were provided and instantly shifted to the steel plates to be hardened and primary cured. In this study, different compositions were defined by fixing the cement content at 10 %wt. and variation of the cellulose pulp content (5, 7.5, and 10 %wt.). Figure 4.5 exhibits the flow chart of the different steps of production and characterization of the composite.

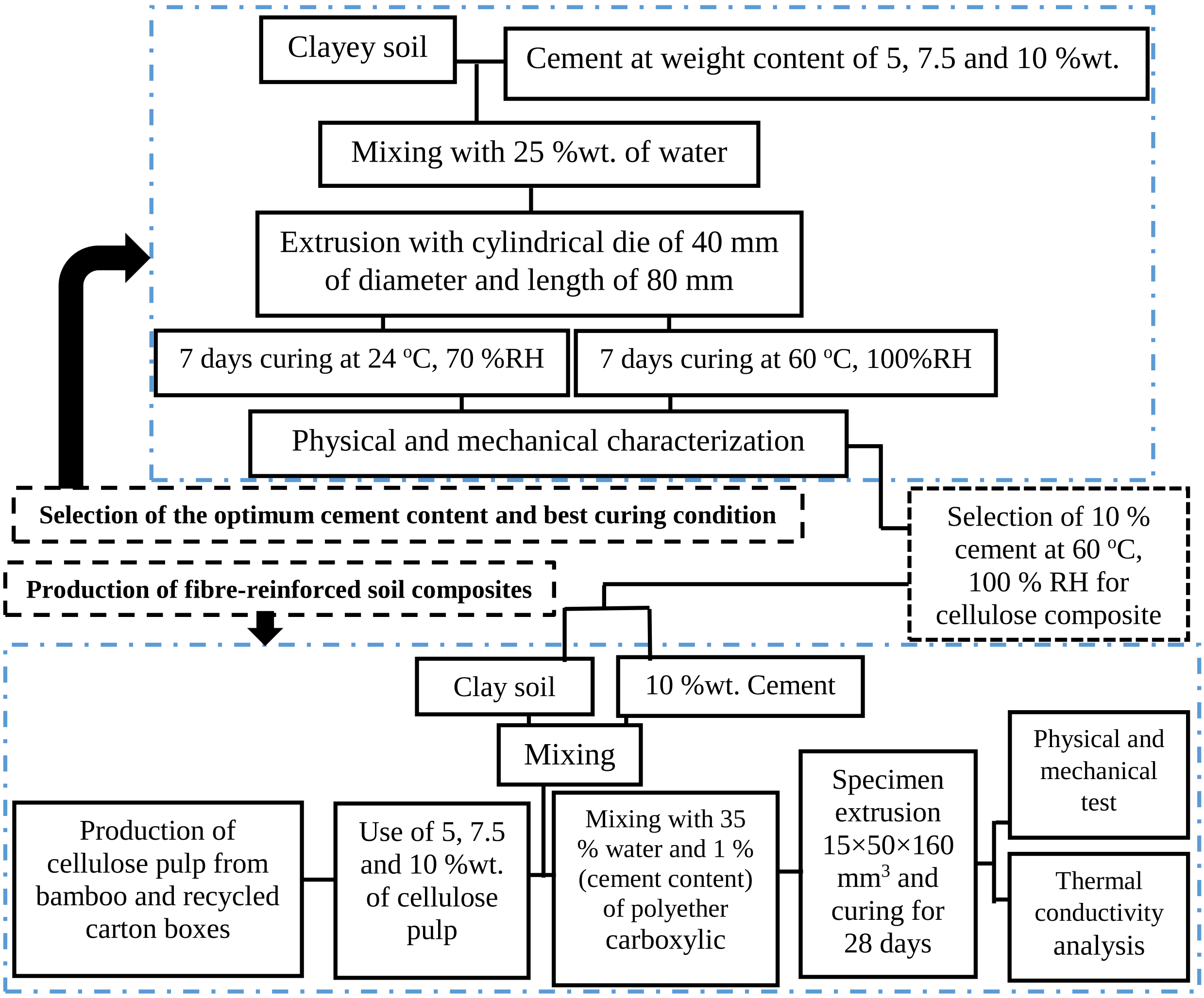


Figure 4. 5 Flow chart of preparation and characterization of the composite

4.2.3.3.Curing condition:

The cellulose composites samples were immediately wrapped after the extrusion with waterproof plastic bags and stored within the laboratory environment for 2 days before transferring into the curing chamber (temperature of 60 ºC and 100 % RH).

4.2.4. Physical characterization

Water absorption, apparent void volume, and bulk density of the earth-based composite were obtained from the average of 6 samples per formulation according to the procedures described by the ASTM C 948 Standard (Testing & Materials, 2009).

The influence evaluation of the inclusion of the fibre on the drying shrinkage was carried out according to the Australian Standards AS1012.13 (Australian Standards, 1992) with a little modification. To ensure a minimum variation in the point of contact with the dial indicator pins and the support, the stainless steel discs with concave fit were glued with epoxy glue to the ends of the specimen (160 mm×50 mm×15 mm) (Figure 4.6b). The measurements of the length and moisture variation were carried out with a digital dial indicator from Mitutoyo, Japan with an accuracy of 1 µm and a digital balance Ohaus adventurer, Germany with an accuracy of 0.01 g respectively. Figure 4.6a presents the experimental setup adapted for the measurement. After 28 days of curing, the samples were directly immersed underwater for 24 h before the first reference measurement. The first measurement of the specimens saturated with water was considered as the reference for the calculation of the shrinkage during drying using equation (1). Then the specimens were placed in the controlling condition with the parameters adjusted to (23 ± 2) °C and (50 ± 5) % RH and measurements were taken daily to verify the variation in length due to the shrinkage of the drying composite for 15 days. Moisture loss ΔW was after drying time t was determined using equation (2).

$$D_s = \left(\frac{C_0 - C_t}{l_0}\right) \times 100 \quad (1)$$

where D_s is the drying shrinkage (%), C_0 is the initial (reference) length (mm) of the saturated specimen, C_t is the daily measurement of the sample length (mm) maintaining in the controlling chamber, and l_0 the length of the specimen measured in mm.

$$\Delta W = \frac{W_0 - W_t}{W_0} \times 100 \quad (2)$$

where W_o and W_t were the starting (reference) moisture content (g) and moisture content (g) after time t of drying, respectively.

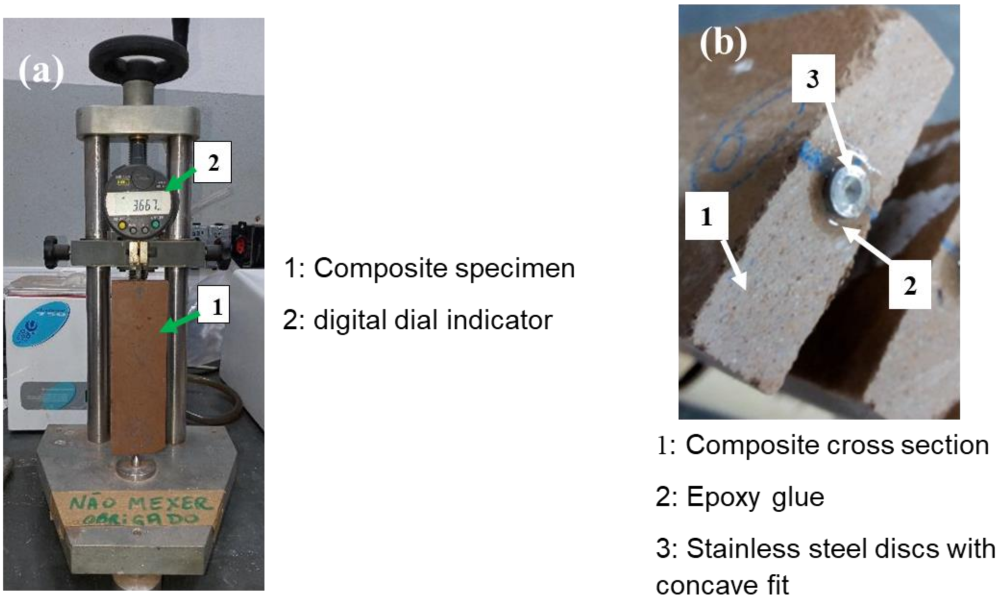


Figure 4. 6 (a) Set-up of the drying shrinkage measurement and (b) cross-section of a composite

4.2.5. Mechanical characterization

Mechanical tests of composites were performed using Emic DL-30,000 testing machine equipped 5 kN load cell for the flexural test. For each fibre content mixture, four-point bending configuration (Tonoli et al., 2007) was used to determine the modulus of elasticity (MOE), modulus of rupture (MOR) and the specific energy (SE). A support span and load span of 135 mm and 45 mm respectively were adopted using a deflection rate of 1 mm/min in the bending test (Ojo et al., 2019). According to Walker (2004), the moisture content impact on the mechanical characteristics of soil materials should be evaluated before testing. Cellulose composites experimented in two conditions: the dry condition in which the specimens were exposed to oven drying at 60 ºC for 24 h before testing. Another set of experiments were performed under the saturated condition where the samples were entirely soaked in water for 24 h. Six samples were tested to four-points bending tests. Equations (3) and (4) define MOE and MOR,

$$\mathrm{MOR} = \frac{\mathrm{P}\,L}{bd^2} \tag{3}$$

$$\mathrm{MOE} = \frac{276L^3}{1296bd^3} \times \left(\frac{P}{\delta}\right) \tag{4}$$

where P is the maximum load (N), L is the span between the supports (mm), δ is the deformation (mm) of the specimen and b and d are the width and thickness (mm), respectively.

The SE was described as the work conducted in the flexural experiment, which is divided by the sample cross-section area (Correia et al., 2014).

The fracture surface after the mechanical tests was analysed applying an optical microscope (Digital Microscope, China) connected to a computer system (Ojo et al., 2020).

4.2.6. Thermal conductivity

The composite thermal conductivity test was carried out according to ASTM E1530 Standards (ASTM Standard & others, 2011), using a heat flow meter DTC 300 (TA Instruments, USA). At least three round samples (diameter of 50 mm and 14 mm thickness) per formulation were cut and machined from the prismatic sample. The cylindrical specimen surfaces were ground and polished for reaching enough smoothness to reduce the friction at the interfaces of the samples with the measuring equipment. Samples were oven-dried at 60 ^{o}C for 24 h before testing. The paste of silicone thermal compound was used to allow reproducible pneumatic loading among the machine sheets and the specimen.

4.2.7. pH evaluation of cement stabilised matrix

The pH of the cement stabilised matrix was determined over time to evaluate its evolution and the potential attack of the cellulose fibre by following the methodology described by (Mármol et al., 2016). 10 g of unreinforced powder (90 %wt. clay soil + 10 %wt. cement), was transferred in 100 ml of deionized water and stirred for 30 min before testing. The pH measurements were carried out after 1, 7, 14 and 28 days, using a pH meter, model Digimed, DM-23, Brazil.

4.2.8. Statistical analysis

ANOVA analysis was employed to ascertain the significance of fibre content on the physical, mechanical and thermal characteristics of the composites. One-way ANOVA and Tukey's HSD tests were applied for determining whether there are distinctions among population means acquired at differing fibres content for each type of fibre.

4.3.Results and discussion

4.3.1. Cellulose fibre properties

The higher percentage of produced bamboo pulp has the width and length ranging from 0–19 µm and 0.6– 1.1 mm, respectively and from 0-19 µm and 0.3-0.8 mm for recycled waste carton pulp fibres (Figures 4.1a and 4.1b). This wide dispersion of the morphology of the pulp fibres is a characteristic of cellulose fibres, which may induce heterogeneity in the reinforcement capacity of fibres and may assist the compression of fibres inside the composite and improve the anchoring between the fibre and matrix (Correia et al., 2014).

The yield of *Bambusa vulgaris* organosolv pulp was 55.6 %wt. and indicated the effectiveness of the pulping process. This is consistent with the results obtained by Ciaramello (1970) which showed respectively 50.3 % and 55.0 % using soda and sulphate pulping of *Bambusa tuldoides*. Similarly, Correia et al. (2015) obtained 51 % from organosolv pulping of *Bambusa tuldoides*. Moreover, the value of the specific density of bamboo pulp (1.558 g/cm^3) was similar to recycled waste carton pulp (1.566 g/cm^3) and was compared well with the mean reported using conventional pulping methods. However, the recycled waste carton pulp presents a higher production yield of 90.8 %wt. compared to bamboo organosolv pulp, which suggests high efficiency of the recycling process.

4.3.2. Water absorption, bulk density and apparent void volume

The introduction of cellulose pulp in the earth-based matrix induce the variation of bulk density with the type and amount of fibre in relationship with the variation of water absorption observed in the unreinforced composite, suggesting a linear relationship between the packing densities of extruded composites and water absorption (Ojo et al., 2019). Similar to the results observed by Correia et al. (2014) on bamboo pulp reinforced cement-based materials, Figure 4.7 shows the enhance of the water absorption and apparent void volume with the fibre content along, with the expected decrease of bulk density. Such behaviour may be due to the hydrophilic character of the cellulose fibre and also a transition region in the fibre–matrix, which is arranged by the water circumambient the fibres that become a poriferous zone when the hydration process is concluded (Correia et al., 2014). These transition zones become more illustrative in the composite volume with the increase of fibre content. In addition, the increase in water absorption capacities is linked to the presence of free hydrogen groups on the surface of cellulosic fibres, which bind to water. The inclusion of recycled waste carton pulp leads to comparative low water absorption, apparent void volume and higher bulk density compared to composite reinforced by bamboo organosolv pulp. This finding may be related to the smaller width of the recycled pulp with improved the fibres packing in the matrix. It may be also linked to the fact that the recycling process may induce the reduction of water retention function of the carton pulp compared to the virgin bamboo pulp. Composites reinforced with recycled cardboard pulp have a water absorption value of less than 37%, which corresponds to the maximum value established for fibre cement by Brazilian Standards NBR 5640 (ABNT NBR.5640, 1995). Only the composite reinforced with 5% bamboo pulp meets this requirement.

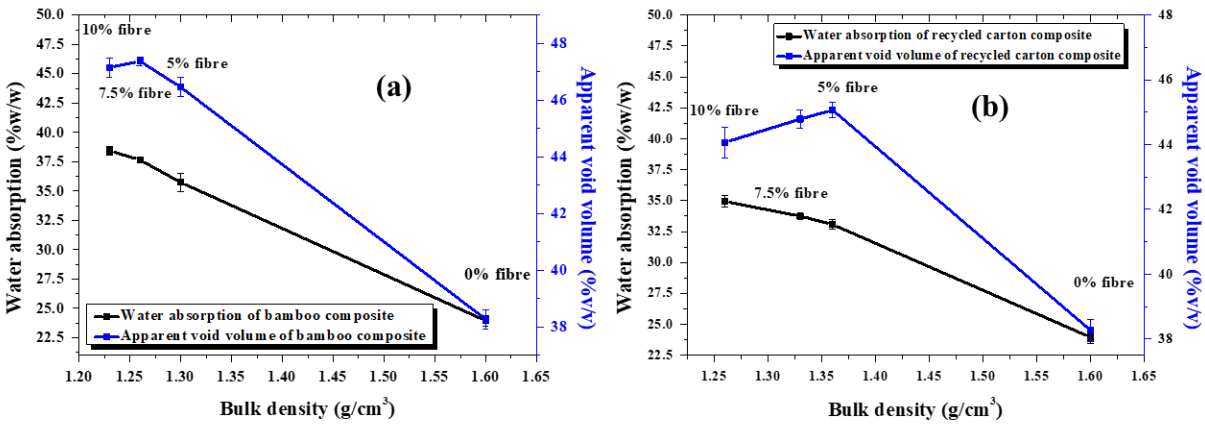


Figure 4. 7 Influence of (a) bamboo cellulose pulp and (b) recycled waste carton pulp on the water absorption, bulk density and apparent void volume of cement stabilised clayey soil matrix at the age of 28 days after mixing.

4.3.3. Drying shrinkage

One of the essential features of earth-based material, which can be used to define its application, is the drying shrinkage. Generally, an acceptable value of drying shrinkage of brick material should be below 8 % (Weng et al., 2003). From the results of Figures 4.8, it has been observed that the attendance of fibres influences significantly in the amount of moisture loss and drying shrinkage. According to De Souza (2014), the variation in the surface tension of colloidal particles and the loss of interlaminar water from the hydration of cement constitute the main mechanisms of drying shrinkage. The drying shrinkage increased with the increase of the amount of cellulose pulp in the soil matrix (Figure 4.8a). The inclusion of cellulose fibre also interferes in the time taken for stabilization (dimensional and mass). The higher dimensional variation observed on reinforced composite may be due to the expansion of fibres during saturation. This decrease in volume of the earth-based composite can be due to the variation in capillary pressure during the drying process. According to Ghavami et al. (1999), natural fibre is prone to absorb water and swell during the mixture of fibre-reinforced soil composite. This expansion disperses the matrix at the micro-scale, and after the drying procedure, the loss the fibres moisture, shrinking to their original size, and leave very small cavities nearby them. From Figure 4.8c, it can be seen that cellulose fibre behaves as a water reservoir of the composite. This function may be associated with its capillary pores and consequently, a higher intrinsic capacity for drying shrinkage. Although the inclusion of cellulose fibre increases the drying shrinkage and the moisture loss of the composite, there is no visible crack on their surface (Figure 4.9b) and all the specimens complied with the requirement (lower than 8 %). Figure 4.9a shows that unreinforced specimen presents some visible crack on their surfaces compared to specimens reinforced by recycled waste carton pulp. This behaviour was supported by (Chiang et al., 2009; Kadir et al., 2016) that a higher amount of fibre contributes to increasing the shrinkage of fired brick material.

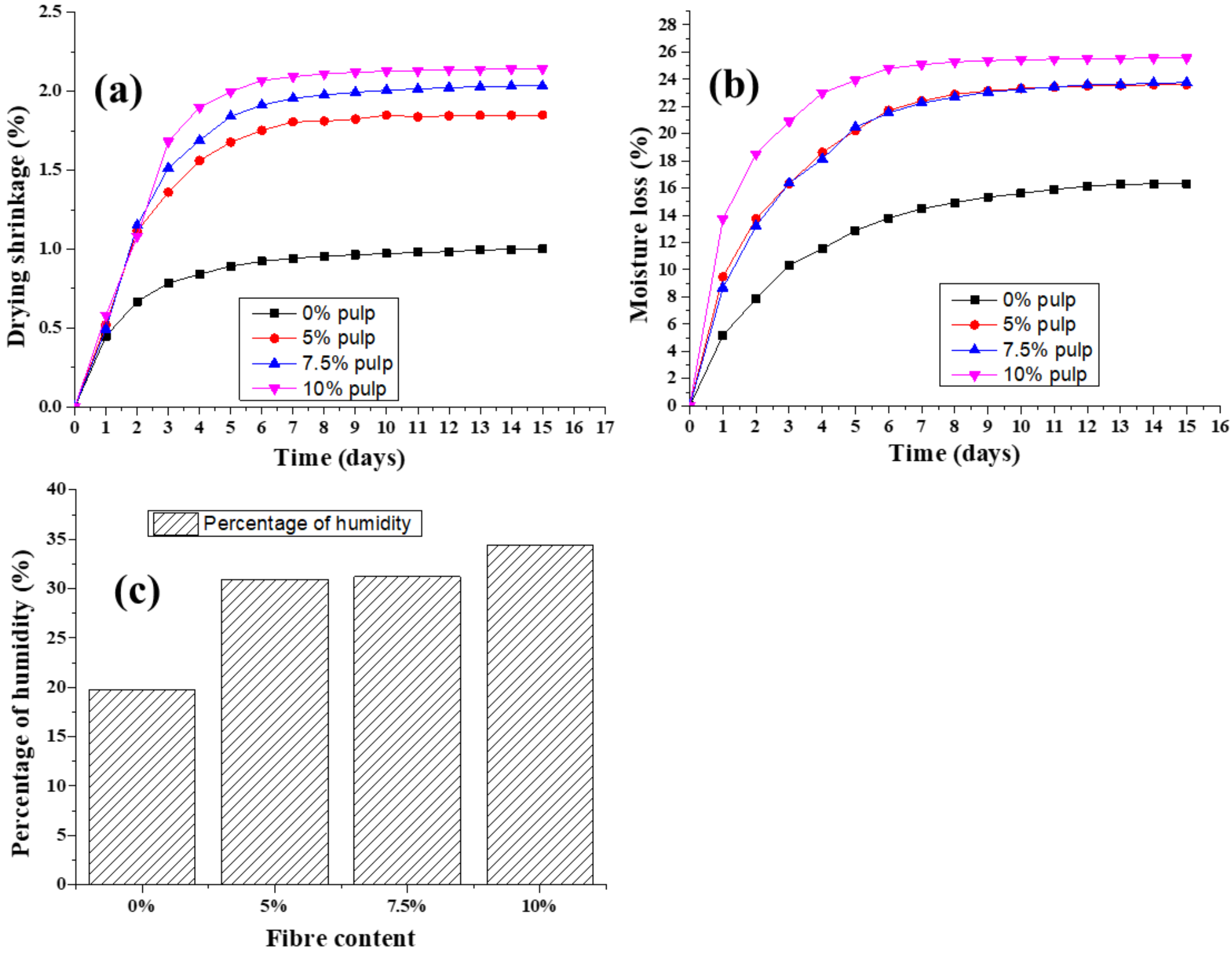


Figure 4. 8 Effect of recycled waste carton pulp inclusion on the (a) drying shrinkage, (b) moisture loss and the (c) percentage of humidity of cement stabilised earth-based composites.

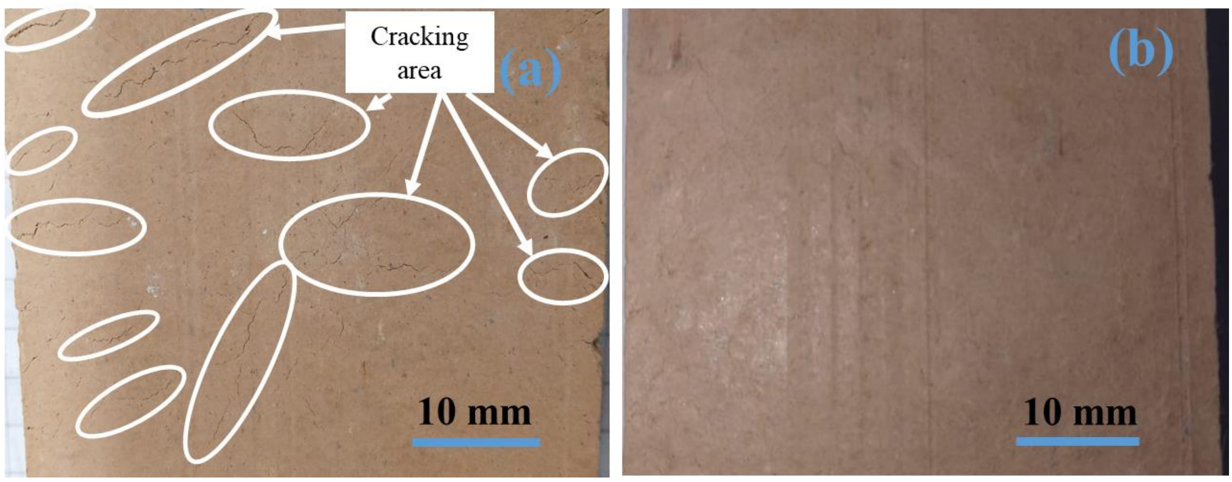


Figure 4. 9 Surface of (a) unreinforced specimen (b) 5 %wt. recycled waste carton pulp reinforced cement stabilised earth-based composites after drying shrinkage test.

4.3.4. Mechanical characterization

In fibre reinforced composite, fibre is usually used to prevent the crack formation in the stress stage, and bridge micro-cracks to expand (Mostafa & Uddin, 2016). The control samples were provided from the same clay soil with the same moisture content, and 10 %wt. cement added. Figure 4.10 presents the cellulose pulp impact on the improvement of the mechanical performance of soil composite. The result revealed a sudden failure of the unreinforced specimen, while the failure of the pulp fibre enhanced earth-based material exhibited multiple cracking (Figures 4.11a and 4.11b) with a progressive breakdown like malleable materials that matches property with other studies (Danso et al., 2015; Ojo et al., 2020). Besides, the inclusion of cellulose fibres restricts the extend of cracks in the earth matrix, as fibres form bridges across cracks before failure (Figures 4.11d and 4.11e) and therefore contribute to the strength improvement (Danso et al., 2015). Moreover, the curves (Figure 4.10) displayed a notable increase in the maximum specific deformation and a small increase of the ultimate stresses of the fibre-reinforced composite over the unreinforced specimen. In addition, the composite reinforced with 5, 7.5 and 10%wt. cellulose pulp fibres develop higher tenacity (area under curves) compared to unreinforced composite (figure 4.9), showing strain hardening behaviour. Then after the first point of cracking (Limit of Proportionality), the stress increases continually. These results may be due to the presence of cellulose pulp which absorbed energy and carried a lot of tensions (Mostafa & Uddin, 2016). It can be concluded that the inclusion of bamboo and recycled waste carton fibres improve ductility and energy absorption of earth-based composite, and has the potential to expand the application of earth-based building materials where specific energy may also be a performance requirement. Therefore, it is clear that the inclusion of more than 2% of cellulose pulp fibres in extruded earth-based matrix ensures satisfactory homogeneity within the material. Besides, it helps to improve flexural strength and ductility of the composite as suggested by Ojo et al. (2019) and reported in fibre cement composites (Correia et al., 2014).

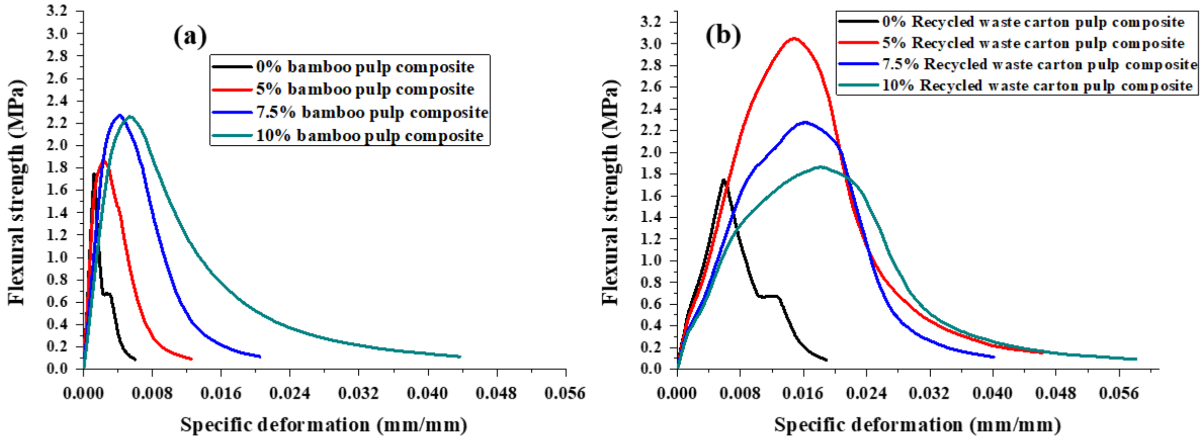


Figure 4. 10 Typical stress-strain curves during the flexural test of the composites containing 0, 5, 7.5 and 10 %wt. of (a) bamboo organosolv pulp and (b) recycled waste carton pulp after 28 days of curing

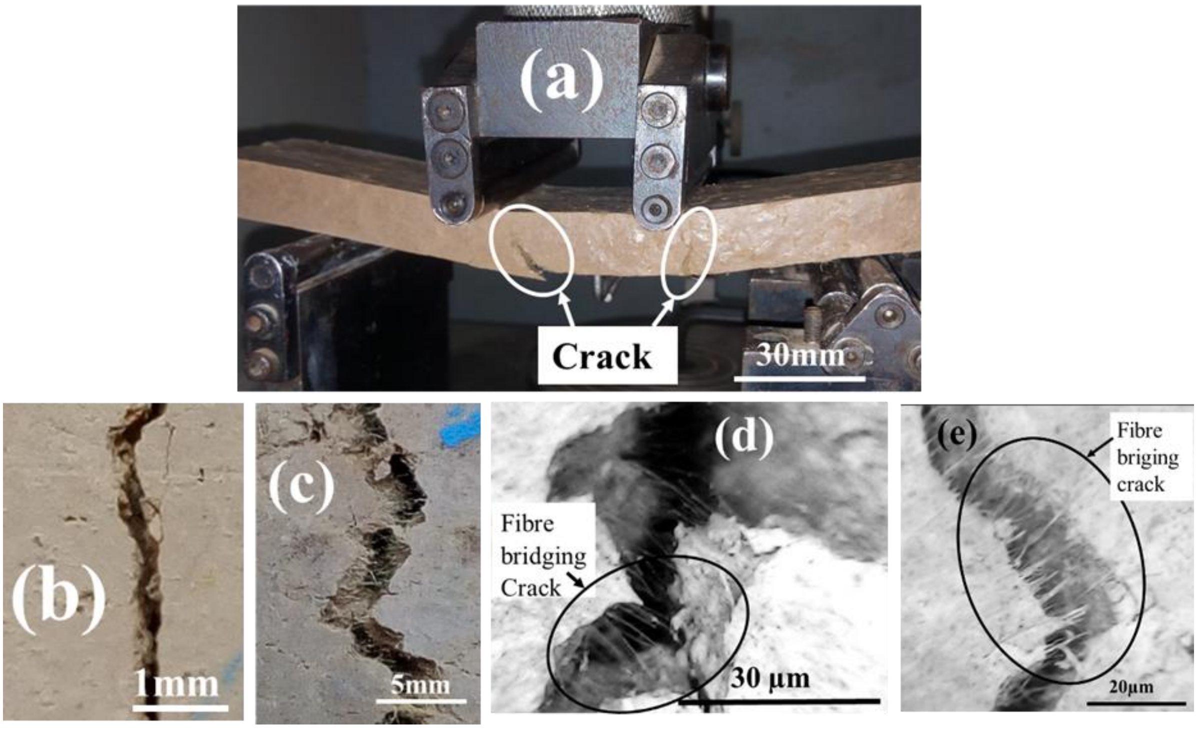


Figure 4. 11 Earth-based composites under flexural test, Failure of (b) unreinforced and (c) cellulose pulp reinforced soil composite and fracture surface of (d) bamboo organosolv pulp and (e) recycled waste carton pulp composite during failure.

Recycled waste carton pulp composite exhibits higher flexural strength before cracking and specific deformation corresponded with maximum stress at 5 %wt. of reinforcement in comparison to 7.5 and 10 %wt. (Figure 4.10b). Whereas, bamboo organosolv pulp showed highest mechanical performance at the weight ratio of 7.5 % (Figure 4.10a). Table 4.3 presents the mean value and standard deviation associated with MOR, MOE and SE of composite reinforced with bamboo and recycled carton pulp. In general, the composite reinforced with both fibres presented higher values of MOR and SE than that controlled at 28 days of curing. In agreement with other studies (Correia et al., 2014; Savastano et al., 2003), MOE presented in Table 4 decreases with increasing of pulp fibre content. This increase of MOR and SE could be explained by the strong interfacial bond among cellulose fibre and soil matrix (Danso et al., 2015). In addition, the reduction of the MOE is related to the transition from the pure brittle unreinforced matrix to ductile fibre reinforced composites (Figure 4.10a). Statistical comparison between recycled waste carton pulp and bamboo organosolv pulp fibre revealed the enhance in flexural strength from unreinforced soil composite to the optimum of 56 % and 39 %, respectively for recycled waste carton, and bamboo pulp reinforced soil materials at dry condition. While the increase at saturated condition was observed at 40 % and 24 %, respectively for recycled waste carton and bamboo pulp reinforced soil composite. Although bamboo organosolv pulp has a high aspect ratio compared to recycled carton pulp (Table 4.1), the inclusion of recycled carton pulp on the soil matrix improves the MOR better compared to bamboo organosolv pulp fibres (Table 4.4). This high strength resulted from the inclusion of recycled pulp should be related to the high friction among fibre-soil matrix (Danso et al., 2015). It can also be explained by the good dispersion due to its small diameter, which fits adequately in the close-packed fabrication of the inorganic matrix, and contributes to a compact uniform microstructure, providing an acceptable balance of mechanical and physical properties of the composite (Peters et al., 2016). An additional explanation of this behaviour may be the higher strength of recycled pulp that is resulting from the Kraft process of the initial wood materials (Monga, 2017). In Table 4.4, it can be seen after a critical point, the strength decreases due to pulp augmentation. It could be explained by the fact that fibres start to tie each other (Ismail & Yaacob, 2011), ending in a decrease cohesion in earth matrix , and inducing weaker fibre-matrix composite. The MOR of 7.5 and 10 %wt. recycled waste carton pulp content was lower than that of 5 %wt. fibre content due to the excessive porosity resulted from the fibre inclusion (Correia et al., 2014), while SE was higher in the post-cracking conditions. However, all the composite flexural strength is higher than the minimum required value establish by ASTM C140 (C140-03, 2011), which is 0.25 MPa. This result suggests the potential application of this composite as a wall construction material.

Statistically, the comparison between the MOR obtained in the dry and wet conditions of the unreinforced matrix revealed a significant drop in average MOR value (wet MOR to dry MOR ratio = 55 %). The water saturation of the composite decreases significantly the mechanical characteristics of the earth-based materials and this can be contributed to the decreasing of soil suction, and a gradual suppression of the bonding force developed through the bridge system (Ojo et al., 2019), weakening the matrix. The MOR of the bamboo pulp fibre reinforced composite is similar to the result (0.9-2.25 MPa) reported by Kolawole et al. (2017) on earth block reinforced with bamboo fibre. Despite the significant drop, the mean value (0.87 MPa) of MOR obtained from saturated unreinforced compared well to the typical value 0.13 and 1.67 MPa reported in the literature for earth-based construction (Galán-marín et al., 2010; Walker, 1995), suggesting good features of the material for masonry block.

SE of the cellulose fibre reinforced earth-based matrix is important since it is related to the reinforcement capacity of fibre in the matrix and also reflects the capacity of transferring load between fibre and matrix (Ballesteros et al., 2019). Table 4.3 shows the increase in SE as the cellulose content increases, and a higher improvement was achieved from recycled waste carton pulp (834 %), in comparison with bamboo pulp composites (372 %) in dry condition. While the increase for the saturation condition was found to be 2167 % and 977 % for recycled waste carton and bamboo pulp fibres reinforced earth-based materials respectively. Thus, the inclusion of cellulose fibre significantly changed the mode of failure of the earth-based matrix from brittle to ductile fracture when subjected to the bending test. This outcome can be attributed to the improvement in the dispersion and alignment of the fibre in the earth-based matrix through the extrusion process, and therefore, help to improve the transfer of stresses between the fibre and the matrix. The required condition for fibres in the brittle matrix is their capacity to enhance the post-cracking behaviour of the composite. Although 10 %wt. of cellulose pulp exhibited that performance, 5 %wt. of recycled carton pulp and 7.5 %wt. of bamboo organosolv pulp content composites were chosen to be evaluated to classify their quality based on ASTM C1186. Standards., (2012).

Table 4. 4 Variation of the MOR, MOE and SE from bending test of bamboo pulp and recycled waste carton pulp reinforced earth-based composite testing on dry and saturated condition after 28 days of curing.

Type of fibre	**Testing condition**	**Fibre content (%wt.)**	**MOR (MPa)**	**MOE (GPa)**	**SE (kJ/m^2)**
Control	Dry	0	1.6 ± 0.2a	2.7 ± 0.2a	0.07 ± 0.01a
	Wet		0.9 ± 0.1	2.4 ± 1.0	0.01 ± 0.01
	Dry	5	1.7 ± 0.3a	1.2 ± 0.1bc	0.27 ± 0.04ab

Bamboo pulp	Wet		1.1 ± 0.1	1.6 ± 0.1	0.13 ± 0.01
	Dry	7.5	2.2 ± 0.1a	1.1 ± 0.1bc	0.28 ± 0.06b
	Wet		1.1 ± 0.1	2.1 ± 0.1	0.13 ± 0.01
	Dry	10	2.0 ± 0.3a	1.2 ± 0.1bc	0.33 ± 0.08b
	Wet		1.0 ± 0.1	1.2 ± 0.3	0.16 ± 0.08
Recycled carton pulp	Dry	5	2.4 ± 0.5a	1.4 ± 0.1b	0.50 ± 0.13c
	Wet		1.2 ± 0.2	1.5 ± 0.1	0.34 ± 0.02
	Dry	7.5	2.1 ± 0.1a	1.4 ± 0.3cd	0.51 ± 0.07c
	Wet		0.9 ± 0.1	0.9 ± 0.2	0.23 ± 0.05
	Dry	10	1.8 ± 0.3a	0.6 ± 0.2d	0.66 ± 0.07c
	Wet		0.5 ± 0.2	0.6 ± 0.4	0.21 ± 0.05

*Value with different letters in the same column have a statistical difference from Tukey post-hoc test ($p < 0.05$)

4.3.5. Thermal conductivity

The thermal conductivity coefficient defined the capacity of the material for conducting heat energy. It is one of the most important thermal insulation properties of building materials (Bentchikou et al., 2012). Figure 4.12 presents the thermal conductivity of pulp fibre composite verse the fibre content (%wt. ratio). The results revealed that the thermal conductivity decrease with increasing the percentage of cellulose pulp in the earth-based materials. Similar behaviour has been reported in other publications in which the authors used different types of lignocellulosic fibre (Bentchikou et al., 2012; Khedari et al., 2001). The observed reduction of thermal conductivity comparatively to unreinforced composite was found to be 26.43 %, 28.66 %, and 33.13 %, respectively for 5, 7.5, and 10% content of bamboo organosolv reinforced clayey soil matrix, whereas in the composite reinforced with recycled carton pulp the reduction was found to be 21.09 %, 36.35 %, and 33.44 %, respectively for 5, 7.5, and 10 %wt. content of fibre. This performance may be explained by the insulation features of vegetal fibres as they have a low thermal conductivity due to their porous structure (presence of lumen) (Robert et al., 2016). Another explanation may be the effect of porosity which occurs in the fibres packing as induction of air bubbles during the mixing operation (Bentchikou et al., 2012). It can be concluded that the inclusion of cellulosic fibres up to 10%wt. in earth-based material significantly diminishes the composite thermal conductivity due to the high porosity, suggesting its application in passive design building (Bidoung et al., 2016). Therefore, consistent with the mechanical performance (Table 4.3), this material can be used to make earthen wall blocks, where flexural strength, ductility, and thermal insulation performance are the main requirements.

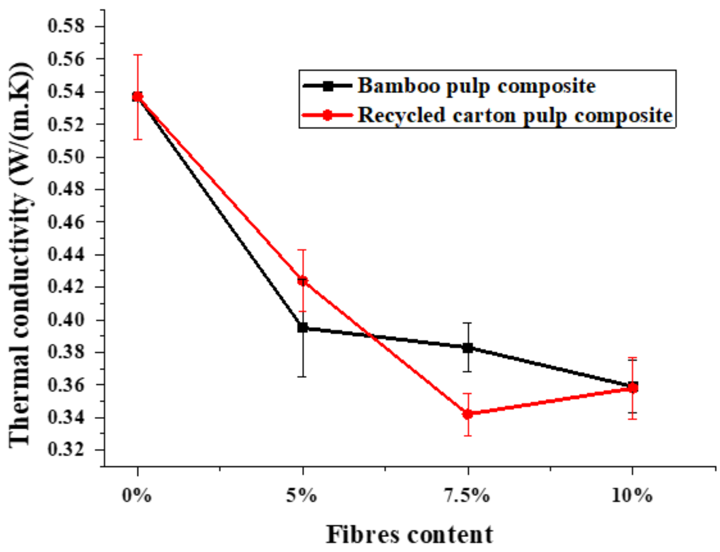


Figure 4. 12 Thermal conductivity value of bamboo and recycled waste carton pulp fibre composite

4.3.6. pH evaluation and cellulose pulp reaction

From Table 4.5, it can be seen that the value of pH of the matrix slightly decreases with time due to the dissociation of the hydration products of cement (such as calcium hydroxide), which are responsible for the high alkalinity (pH > 11.62). According to Tolêdo et al. (2000), porous alkalinity medium (pH > 11), causes a progressive degradation of cellulose fibre and induces the reduction of its reinforcing performance. This result suggests the need for applying the carbonation process on the composite to reduce the pH of the composite and in consequence, raise the durability of the cellulose fibre.

Table 4. 5 pH evolution over time of cement stabilised matrix

Time (days)	**0**	**1**	**7**	**14**	**28**
pH value	12.16	12.04	11.92	11.89	11.62

4.4.Conclusion

This Chapter presents an experimental investigation of a novel application of cellulose pulp fibre in earth-based construction to give insight on the effect of recycled waste carton and bamboo organosolv pulp, and dosage on physical, mechanical, and thermal conductivity features of cement stabilized clay soil matrix. Recycled waste carton pulp fibre is a promising alternative reinforcement of earth-based building material, due to its availability, its short-term renewal, and its easy process along with a high yield of production. According to the results described above, the subsequent conclusions can be expressed:

- Inclusion of recycled waste carton pulp in earth-based materials significantly increases the moisture loss, the drying shrinkage, and behaves as a water reservoir for the composite. Thus there is a need to apply a treatment on the fibres to improve their dimensional stability and reduce their water retention capacity.
- By increasing the cellulosic fibre content, bulk density decreases, but water absorption and apparent void volume increase. This conclusion revealed that the transition area in the fibre–matrix becomes increasingly representative in the volume of the composite when the fibre content increase.

- As bamboo pulp content increases, the MOR and SE increase, while the MOE decreases during the bending solicitations. The optimum improvement in MOR was found to be 39% and 24% respectively at the dry and wet conditions at 7.5%wt fibre content in comparison to the control sample, and the optimum SE was achieved at 10 %wt. fibre content with an increase of 372 % and 977 % respectively at the dry and wet condition.
- The recycled waste carton pulp fibres insertion in soil composite improved the flexural strength of the composite by increasing MOR between 13 % and 56 % and SE between 623 % and 834 % in dry condition compared to unreinforced composite. While the saturated condition presents an increase in MOR of 40 % and SE of 2167 % at 5 %wt. of fibre content, which suddenly decreases with the addition of reinforcement.
- The minimum value of the modulus of rupture obtained from an unreinforced composite tested in the dry state was 1.6 MPa, which is within the typical range of values obtained in the literature (0.13-1.67 MPa) for unfired clay, suggesting the potential of the cellulose fibre reinforced earth material as well as an extrusion moulding mechanism for the production of earth composites.
- The summation of cellulose pulp fibres yields a better thermal insulation behaviour of soil matrix, as at 7.5 %wt. recycled waste carton fibre content, k = 0.342 W/(m.K) which corresponding to 36.35 % of reduction compared to the control sample.
- Earth-based composites reinforced by recycled waste carton fibres from the post-consumer waste present more advantages of resources and energy conservation and waste reduction as they can be used for construction and insulation. In addition, the successful replacement of virgin bamboo pulp fibres with recycled waste carton pulp fibres reduces the environmental footprint of the building material.

Physical, mechanical and thermal behaviour of composite reinforced with 5 %wt. of recycled waste carton pulp and 7.5 %wt. of bamboo organosolv pulp fibre suggest a suitable utilization of these cellulose pulp fibres as a lightweight building material in all countries where there is a pressing need for a housing solutions to solve both energy and environmental concerns.

The durability to the wetting and drying cycle of the composite and the ability of the cellulose fiber to stop the propagation of cracks in the composite under a three-point bending load (Fracture toughness) are assessed in the following chapter.

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CHAPTER FIVE

5.0.Performance and durability of cellulose pulp reinforced extruded earth-based composites[4]

5.1.Introduction

Recycling is an important strategy for the sustainability of waste management systems because of its economic and environmental benefits (Ibáñez-Forés et al., 2018; Marques et al., 2014). Over the past decade, the approach to recycling packaging waste has been of great concern around the world due to its environmental impact (Marques et al., 2014). Furthermore, the increase of environmental awareness nowadays, promote the use of cellulose wastes as an additive in the construction industry (Bentchikou et al., 2012; Marikunte & Soroushian, 1995; Soroushian et al., 1996; Soroushian & Shah, 1993). Bentchikou et al. (2012) reported that 4% of recycled fibre from waste paper and packaging enhanced the flexural strength and reduce the bulk density and thermal conductivity of the cement-based composite. According to the World Bank report (Kaza et al., 2018), the generated municipal solid waste in 2016 was estimated at 2,010 million tonnes and paper and cardboard represented 17 % of this global waste. The prevision of this global waste is evaluated at 3,500 million tonnes in 2050. Therefore, it is clear that this cardboard and paper waste from municipal solid waste is a promising source of cellulosic fibres, due to its global availability.

Cellulose pulp reinforced cementitious materials show remarkable improvement in fibre-matrix bond, greater reinforcement efficiency, and a significant increase in flexural strength and toughness when compared with the use of vegetable fibre in strand form (Karade,

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2010; Savastano et al., 2003). The wood pulp which is an example of cellulose fibre provides a high specific binding area, mechanical anchorage, and crack-bridging in the brittle matrix due to its high aspect ratio filaments, irregular surface, and low coarseness (Savastano et al., 2004). The homogenous distribution of the pulp fibre within the matrix remains the key parameter to achieve this performance. In addition, the use of cellulose pulp as reinforcement in cement stabilized earth-based matrix can provides additional moisture paths within matrices and consequently improves the cementitious and pozzolanic reactions during curing time. This can be due to their hydrophilic feature, water retention capacity, and porous structure (Ferrara et al., 2015).

The fast growth, relatively low cost, and worldwide availability of bamboo materials along with its strength, constitute a promising fibre source for pulping process and reinforcement in various composites as compared to other vegetable sources (Correia et al., 2015). A study conducted by Correia et al. (2014), showed that the inclusion of 8% of bamboo organosolv pulp in a cementitious matrix, enhanced the flexural strength/ fracture toughness of the composites. Similarly, cellulose pulp such as eucalyptus (Ballesteros et al., 2015; Santos et al., 2015), sisal (Tonoli et al., 2007), and bleached pine (Ballesteros et al., 2015), have been used to improve the mechanical properties of cement-based materials. To date, the evaluation of the properties of the earth-based composite reinforced with cellulose pulp fibres is not well documented. Recently, Ojo et al. (2019), demonstrated that the use of less than 2% by volume of eucalyptus pulp fibres was not sufficient to improve the ductility of the alkali-activated earth material. Furthermore, Muñoz et al. (2020), reported that the introduction of 12.5% paper and pulp into the adobe brick increases the compressive strength to 190% while reducing thermal conductivity by 30%, compared to unreinforced brick. The evaluation of durability against wetting-drying cycles, dimensional stability, and fracture mechanism of an earth-based material reinforced with cellulose pulp is not well known.

With regards to durability in stabilized soil materials, the major concern is the loss of structural integrity when exposed to weathering (wind, rainstorm, and other degrading agents) (Manu, 2013; Ojo et al., 2018). The effectiveness of any stabilization mechanism needs to be examined with a strict durability test to assess the influence of binder against material loss through the life service (Avirneni et al., 2016). This may be due to the variation of the temperature and the infiltration of rainwater in the exposed structure. Given its susceptibility to weathering, the durability over wetting-drying (w-d) cycles of cellulose pulp reinforced soil-building material is a significant criterion for service life evaluation.

In addition to stiffness, strength, and toughness, the performance of building elements also mainly depends on the drying shrinkage of the constitutive materials. Shrinkage is considered as one of the main drawbacks identified in the long-term sustainability of building material because it causes early age micro and macro-cracks (Jiang et al., 2014; Sivakumar & Santhanam, 2007). The mitigation of stress developing during the drying process and the formation of shrinkage cracks is usually ensured by the use of fibre as reinforcing additives. The mechanism of crack growth inhibition is thought to be due bridging action of the fibres. The previous investigation on the durability of soil materials reinforced with lignocellulosic fibres as additives has identified the swelling and shrinkage of natural fibres in drying earth-based material as a major problem (Bouhicha et al., 2005; Danso et al., 2015a; Ghavami et al., 1999; Kampala et al., 2014; Walker, 1995). Studies to understanding the limitation of the application of soil material in the building industry and enhance its life span by introducing additive binder (Kampala et al., 2014; Walker, 1995) such as agro-waste fibres (Danso et al., 2015b), coconut and sisal fibres (Ghavami et al., 1999), and straw (Bouhicha et al., 2005). Sangma & Tripura, (2020), evaluated the effect of natural fibres on the linear shrinkage of the earth liner material. Results show that the addition of natural fibres reduces linear shrinkage, density and crack formation, compared to unreinforced composites. However, the present study attempts to investigate the effect of adding cellulose pulp fibres on shrinkage cracks in an earth-based material and the influence of an alternating wetting-drying cycle on the dimensional stability of the composite.

The improvement of stabilized soil performance has been attributed to physical-chemical interactions between soil, cement, and water (Horpibulsuk, 2012). These physical-chemical interactions can be optimized by controlling the process, curing temperatures and moisture conditions (Maskell et al., 2014). Although compression molding is the traditional processing technique for producing stabilized block, extrusion molding has been successfully used as an efficient processing method in the forming of earth-based pastes for the production of clay bricks (Ojo et al., 2019). Khelifi et al. (2016) demonstrated that the fibres do not significantly improve the mechanical behaviour of cast materials, but in contrast, the extrusion process improves the fibre dispersion and fibre/matrix bond quality and consequently, enhances the mechanical performance of the composite. Ojo et al. (2019) investigated the effect of fibre reinforcement on the properties of extruded alkali-activated clayey soil and revealed the improvement of the mechanical performance of the extruded composite as a result of the high packing density due to the extrusion process.

This Chapter of the thesis evaluates the durability, mechanical, and drying shrinkage properties of viable constructive materials in developing regions using non-conventional bamboo organosolv pulp and recycled waste carton pulp. Specifically, this work: (i) evaluates the effect of varying the fibre weight fraction on the modification of flexural capacity of cement stabilised soil; (ii) examines the change in fracture toughness behaviour induced by fibre the inclusion in the matrix, (iii) evaluates the durability against wetting-drying cycles of cellulose pulp reinforced earth-based material, and (iv) examines the effect of cellulose pulp addition on the repeating drying shrinkage test of the earth-based composites.

5.2.Materials and Methods

5.2.1. Preparation and Characterization of Pulp

Bamboo pulp was obtained from *Bambusa vulgaris* chip using organosolv method described by Stanislas et al. (2020). A concentration of 20g/L of the carton boxes material (manually cut into small pieces) and tap water were prepared and stored for 48 h before transferring to the disintegrator (Marconi, model MA 758, Brazil) to defibrillate for 1 h. Excess water from the mixture was drained manually using 0.044 mm sieve. Similar methods have been used to defibrillate raffia cellulose pulp (Stanislas et al., 2020).

The specific density of bamboo organosolv pulp and recycled waste carton pulp was determined by the Helium gas pycnometer (Quantachrome Instruments, USA). Cellulose pulp fibre microstructure was performed by scanning electron microscope (SEM), using Hitachi TM-300 with a low vacuum. The pulp fibres were analysed without metallic coating with an accelerated voltage of 15 kV and backscattered electron mode using magnifications of x2000 (Ballesteros et al., 2017).

5.2.2. Characterization of the Soil

This soil used in this study was defined in the previous section (chapter 4). According to the previous study of this soil, the mineral composition is dominated by muscovite (52.5%), quartz (32.7%), and sanidine (12.9 %), while the chemical composition is SiO_2 (70.39%), Al_2O_3 (13.31%), Fe_2O_3 (4.09%) and K_2O (4.97%) %) (Ojo et al., 2020; Ojo et al., 2019). The cement used as a stabilizer was Ordinary Portland Cement, type CP V-ARI according to Brazilian Standards NBR 5733 and as per ASTM-C150 Type III Standards, provided by Intercement Brazil. Figure 5.1 presents the particle size distribution of cement and soil using LA-950V2 Partica Particle Size Analyzer. It can be seen in Figure 5.1 that soil showed a coarser distribution, with 75 % of particles < 50 µm, compared to cement with 75 % of particles < 27 µm.

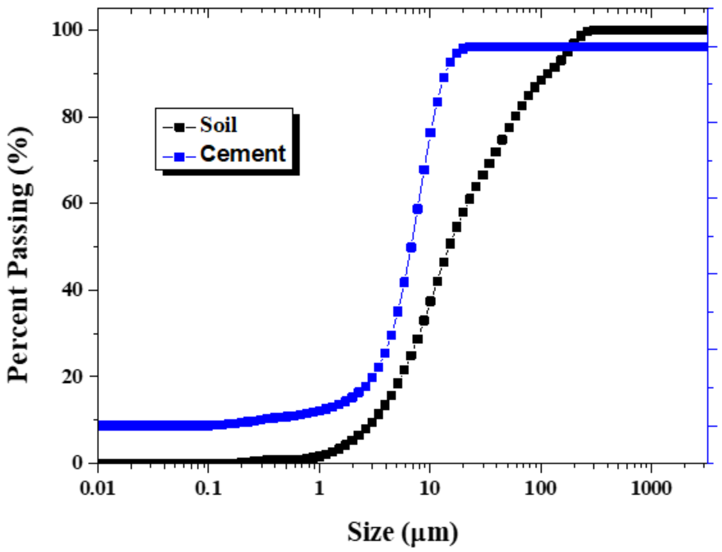

Figure 5. 1 Particle size distribution of soil and cement

5.2.3. Specimen Preparation, Mix Proportion, and Curing Condition

Cement and dry soil were mixed as described in Table 5.1 at high speed before the introduction of the fibres with additional mixing for 10 min. The proportion of 1 %wt. of cement content of water reducer polyether carboxylic (commercial name ADVA 190) was mixed to approximately 35 %wt. of water and used as rheological modifiers to promote pseudo-plastic behaviour of the composite (Santos et al., 2015). The paste was mixed for 5 min before it was transferred to an MVIG-05 model, laboratory extruder (Gelenski Ltda, Paraná, Brazil) with a cross-section die width/height ratio of 3.3 and operating at a linear speed of approximately 4 mm/s. Plates of 160 mm×50 mm×15 mm were produced and immediately transferred to the steel plates for hardening and initial curing. In this study, different compositions were obtained by fixing the cement content at 10 %wt. and the variation of the cellulose pulp content (5, 7.5, and 10 wt.%). Figure 5.2 presents the flow chart of the different steps of production and characterization of the composite. The cellulose composite samples were immediately wrapped after the extrusion with waterproof plastic bags and stored within the laboratory environment for 2 days before transferring into the curing chamber (temperature of 60^{o}C and 100% RH) for 26 days prior test.

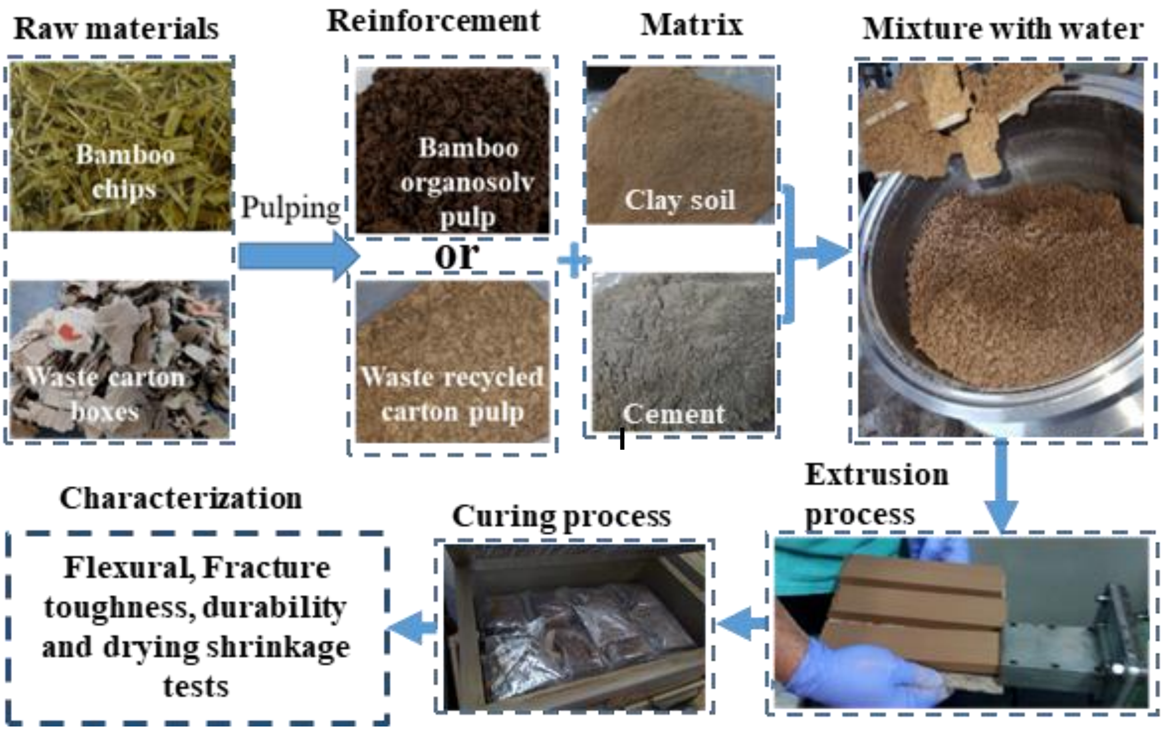

Figure 5. 2 Flow chart of preparation and characterization of the composite

Table 5. 1 Composition of the extruded cellulose pulp reinforced earth-based composites

Type of pulp fibre	Reference	Bamboo organosolv pulp			Recycled carton pulp		
Pulp content (%wt.)	0	5	7.5	10	5	7.5	10
Cement content (%wt.)	10	10	10	10	10	10	10
Soil content (%wt.)	90	85	82.5	80	85	82.5	80

5.2.4. Physical Characterization

Bulk density (BD), water absorption (WA) and apparent void volume (AVV) were obtained from the average of six samples of each formulation by following the procedures specified by the ASTM C 948 Standards (Testing & Materials, 2009). The drying shrinkage of the composite was determined over time to evaluate the effect of cellulose pulp on the dimensional stability and moisture loss of earth-based matrix by following the modified Australian Standards AS1012.13 (Australian Standard, 1992) as described by Giordano et al. (2009). Three cycles of the wetting-drying process were carried out to evaluate the effect of repeating wet-dying cycles on the moisture loss, percentage of humidity and drying shrinkage.

5.2.5. Durability

The durability study based on the wetting-drying cycles was performed on unreinforced and reinforced composites to evaluate the effect of cellulose pulp inclusion on the weathering resistance. The test was carried out on the average of five specimens per formulation according to ASTM D 559-57 (ASTM, 1996). The procedure of wetting in tap water for 5 h and drying at 70^{o} C for 42 h constituted one cycle. Then the samples were weighted after giving six brush strokes on the two horizontal surfaces with a metal wire brush at constant pressure. After 12 cycles of wetting and drying, the wearing performance was evaluated as a percentage of reduction of the initial dry mass.

5.2.6. Mechanical Testing

Mechanical tests were carried out according to the methodology adopted by Santos et al. (2015). A servo-hydraulic mechanical testing machine (MTS Model 370.02, Eden Prarie, MN) equipped with a 1 kN load cell was used to conduct the tests. Prismatic specimens (80 mm x 20 mm x 15 mm) were machined from extruded samples and polished to the sides to aid the observation of cracks/microstructure.

Mechanical tests were conducted under three-point bending with a support span of 55 mm and a loading rate of 1 mm/min. A minimum of five samples were tested for each formulation and the Modulus of Rupture (MOR) and Modulus of Elasticity (MOE) were determined using equation 1-2 (Zweben et al., 1979).

$$\text{MOR} = \frac{3\text{P}\,S}{2BD^2} \quad (1)$$

$$\text{MOE} = \frac{S^3}{4BD^3} \times \delta \quad (2)$$

where P is the maximum load (N), S is the span between the support (mm), B and D are specimen width and thickness (mm), respectively, and δ is the load/deflection slope of the initial straight-line portion (N/mm).

SE is defined as the work conducted in the flexural experiment, which is divided by the specimen cross-sectional area (Ferrara et al., 2015).

5.2.7. *Fracture Toughness*

The evaluation of the resistance of crack propagation in the earth-based matrix was performed by mode 1, fracture toughness, K_{IC}. The Single Edge Notched Bend (SENB) specimens with a notch to depth ratio of 0.45 were adopted to determine K_{IC}. A minimum of five specimens per formulation was subjected to monotonic loading (under three-point bending loading) and a crosshead speed of 15 mm/min was adopted. The fracture toughness was determined from equation 3, according to ASTM E399-12 (ASTM Standard, 2012).

$$K_{IC} = \frac{\text{P}\,Sf(\frac{a}{w})}{BW^{1.5}} \quad (3)$$

$$\text{where } f\left(\frac{a}{W}\right) = \frac{3(\frac{a}{W})^{0.5}\left[1.99-(\frac{a}{W})(1-\frac{a}{W})(2.15-3.93\left(\frac{a}{W}\right)+2.7\left(\frac{a^2}{W^2}\right))\right]}{2(1-\frac{2a}{W})(1-\frac{a}{W})^{1.5}} \quad (4)$$

where P is the maximum applied load (N), B and W are specimen thickness and depth (mm), respectively, S is the loading span, and a is the crack length (mm). This equation was adopted under the assumption that the Linear Elastic Fracture Mechanic (LEFM) conditions prevail (Mustapha et al., 2016).

5.2.8. Microstructural Characterization

Cellulose pulp fibre composite microstructure was performed by the scanning electron microscope (SEM), using Hitachi TM-300. The composites were impregnated in the epoxy resin and curing for 24 h under vacuum before polishing according to the methodology described by Correia et al. (2014). The polished composites were analysed after coating with carbon with an accelerated voltage of 15 kV and backscattered electron mode.

5.2.9. Statistical Analysis

To ascertain the effect of the fibre inclusion on the physical, mechanical properties of earth-based composites, one-way ANOVA tests as well as Tukey's HSD (Honestly Significant Difference) test, were used to determine if there exist differences between population means obtained at varying cellulose pulp content for a specific type of fibres.

5.3.Results and Discussion

5.3.1. Characteristics of Cellulose Fibre

Figures 5.3a and 5.3b show the morphology of bamboo organosolv and recycled waste carton pulp fibres with the diameter range of microscales. Table 5.2 presents the average width, length, and specific density of cellulose pulp fibres. The bamboo pulp fibres width and length were in the range of 4–23 µm and 0.208– 1.734 mm, respectively and for recycled waste carton fibres width and length were in the range of 4-58 µm and 0.372-2.350 mm, respectively. This wide dispersion of the morphology of the fibres is a feature of vegetable fibres which may cause heterogeneity in the reinforcement ability of fibres and further aid the packing of fibres within the composite (Correia et al., 2014)

The production yield of *Bambusa vulgaris* organosolv pulp (55.6 %wt.) suggests an effective pulping process. A similar result has been reported on soda and sulphate pulping process of *Bambusa tuldoides* with the yield value of 50.3% and 55.0%, respectively (Ciaramello, 1970), while Correia et al. (2015) obtained 51% from organosolv pulping of *Bambusa tuldoides*. The waste carton boxes recycling process offers significantly high production efficiency (yield value of 90.8 %wt.) compared to organosolv bamboo pulp, suggesting a promising economic benefit for this recycling process. The specific density is within the mean found in the pulping process carried out by conventional methods (Ojo et al., 2019).

Table 5. 2 Physical and morphological characteristics of cellulosic fibres

Type of fibre	**Width (µm)**	**Length (µm)**	**Specific density (g/cm^3)**	**The yield of production (%wt.)**
Bamboo organosolv pulp	10.75± 3.64	868 ± 469	1.558 ± 0.009	55.6
Recycled waste carton pulp	16.43 ± 8.85	945 ± 417	1.566 ± 0.011	90.8

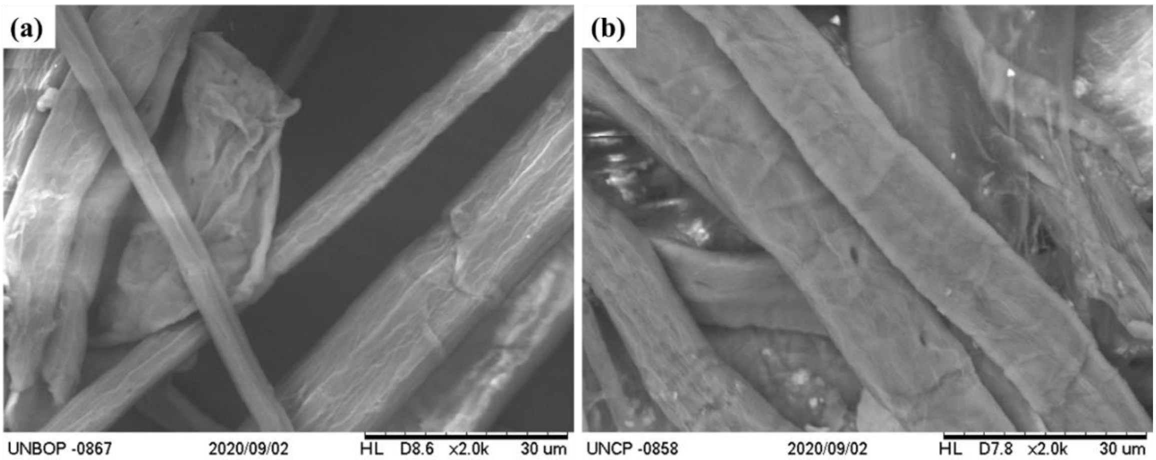

Figure 5. 3 SEM micrographs of cellulose pulp fibre from (a) bamboo and (b) recycled waste carton. X2000 magnification range.

5.3.2. Water absorption, bulk density, and apparent void volume

The density of earth-based building materials is regarded as a significant indicator of strength and durability. As expected, the water absorption and apparent void volume were closely related to the pulp content, significantly increasing with increasing pulp fibres content (Table 5.3). However, the bulk density decreased with increasing cellulose pulp content for both species. These behaviours seem to be the result of the larger amount of lumens (most fibres) of cellulose pulp (Figure 5.4b and 5.4c) along with their high porosity (Ballesteros et al., 2015). Composites reinforced with bamboo pulp had higher water absorption and apparent void volume values (Table 5.3) when compared to recycled waste carton pulp. This can be linked to the fact that the recycling process may have induced the reduction of water retention function of the carton pulp compared to the virgin bamboo pulp. The results showed that the average values of water absorption of unreinforced and recycled pulp composite were below 37% (Table 5.3), in line with the acceptable value suggested by the NBR 5640 (ABNT NBR.5640, 1995), which was achieved only with the composite containing 5 wt.% of bamboo pulp. Furthermore, all the bulk density values fall within the range of lower bound of typical values obtained for earth-based building materials and classified as lightweight construction materials (Ojo et al., 2019).

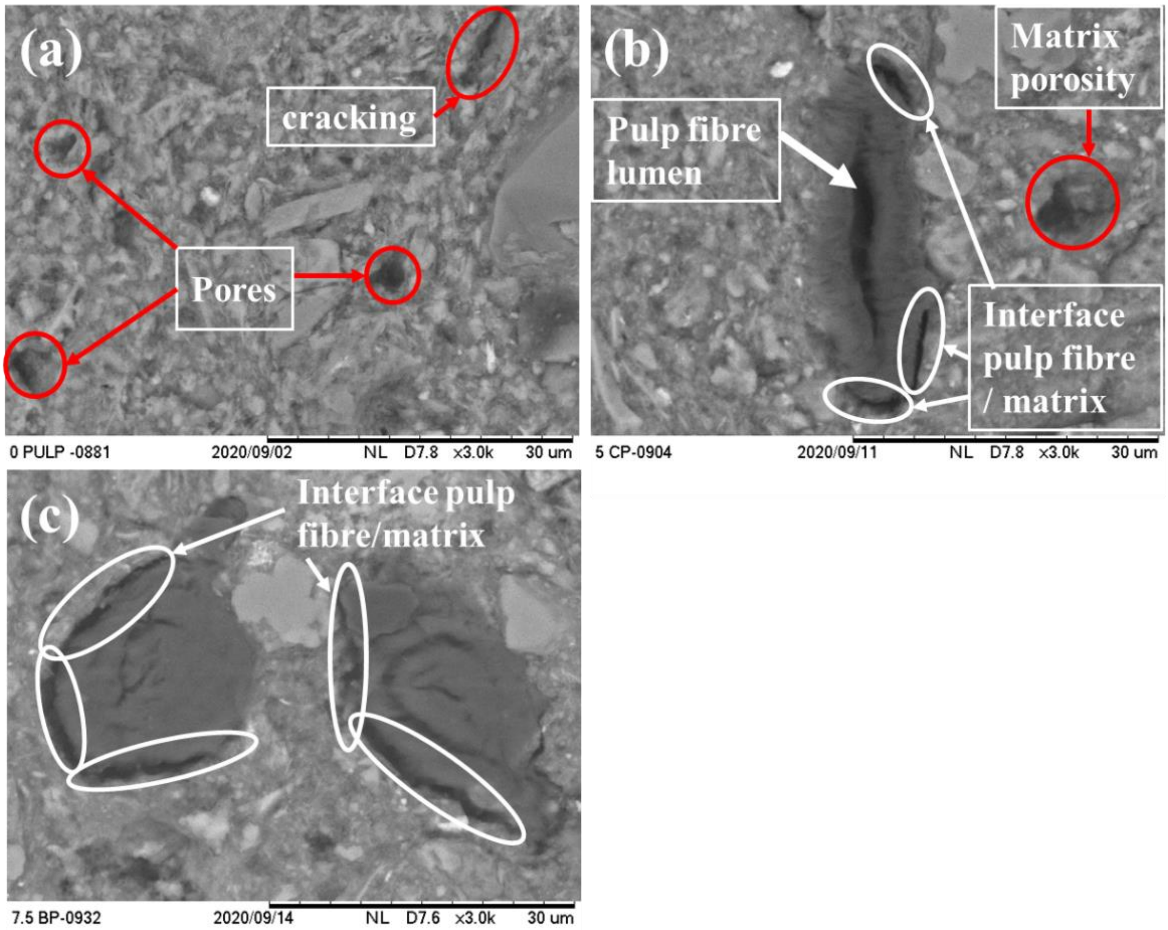

Figure 5. 4 SEM micrographs of (a) unreinforced earth-based composite, (b) composite reinforced with 5 %wt. of recycled carton pulp and (c) composite reinforced with 7.5 %wt. bamboo organosolv pulp with backscattered electron mode (BSE)

Table 5. 3 Physical properties of cellulose pulp reinforced earth-based composites.

Type of fibre	**Pulp content (wt %)**	**Water absorption (wt%)**	**Bulk density (g/cm^3)**	**Apparent void volume (%v)**
Unreinforced	0	23.95 ± 0.47a	1.60 ± 0.02a	38.27 ± 0.34a

Bamboo pulp	5	35.74 ± 0.80d	1.30 ± 0.02cd	46.46 ± 0.33d
	7.5	37.65 ± 0.18e	1.26 ± 0.01de	47.37 ± 0.16e
	10	38.43 ± 0.39e	1.23 ± 0.02e	47.15 ± 0.34de
Recycled waste Carton pulp	5	33.08 ± 0.39b	1.36 ± 0.01b	45.07 ± 0.23c
	7.5	33.75 ± 0.22bc	1.33 ± 0.01bc	44.79 ± 0.29bc
	10	34.93 ± 0.46cd	1.26 ± 0.03de	44.07 ± 0.47b

*Value with distinct letters in the identic column mean statistical difference resulted from Tukey post-hoc test ($p < 0.05$)

5.3.3. Drying Shrinkage

The process of introducing cellulose fibre in soil matrix was adapted to prevent cracking shrinkage which is the major concern in the earth-based block and its resolution provide a quality guarantee for sustainable building component. Figure 5.5 presents the results of a repeated drying shrinkage test of earth-based composite reinforced with recycled carton pulp. The drying shrinkage increased with the cellulose pulp inclusion. This may be attributed to the swelling and shrinkage property of the soil matrix and change in dimension of cellulose pulp fibre in the presence of moisture and temperature variation (Figures 5.4b and 5.4c). Similar behaviour has been reported for other soil composites in which the dimensional change has been attributed to the expansion of the fibre after absorbing water during mixing and shrinkage back almost to the original dimensions with the loss of moisture (Ghavami et al., 1999) leaving space between fibre and matrix (Figures 5.4b and 5.4c). Although the value of drying shrinkage increases (Figure 5.5) from 1.024 to 2.143% with cellulose pulp inclusion, it agrees with the acceptable value of 8 % recommended for brick materials (Weng et al., 2003). There was no visible crack on the surface of cellulose pulp reinforced composite compared to unreinforced composite during the drying process (Figures 5.6a and 5.6b). This implies that cellulose pulp intercept cracks propagation on the surface of earth-based material during the drying process. Figures 5.7a and 5.7b present the effect of cellulose pulp on moisture loss and percentage of humidity, respectively. The results showed that the moisture loss and percentage of humidity increase with an increase in pulp content. This is attributed to the high absorption capacity of pulp fibre lumens (Figure 5.4b). However, unreinforced and reinforced composite showed a lower reduction of the percentage of humidity (Figure 5.7b) and moisture loss (Figure 5.7c) after the second cycle of drying following by rewetting, with an improvement on the dimensional stability (Figure 5.5).

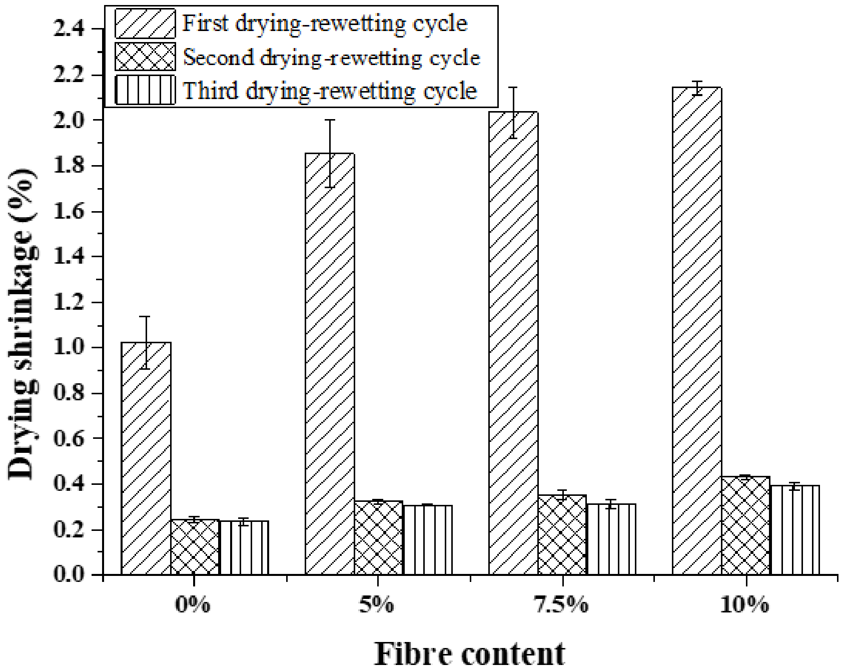

Figure 5. 5 Drying shrinkage reduction after drying following by rewetting cycles of drying shrinkage

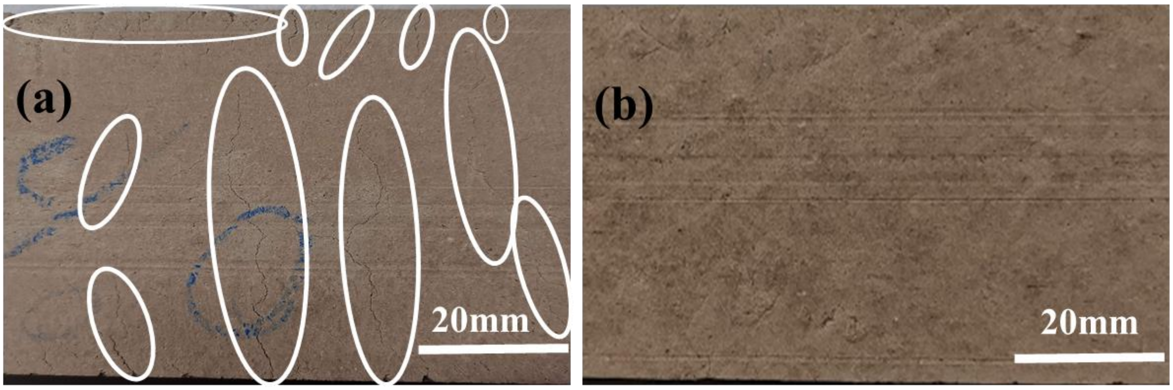

Figure 5. 6 Surface of (a) unreinforced and (b) 5% recycled carton pulp reinforced earth-based composite after three cycles of wet-drying at 23 ºC and 50 % of relative humidity. The circled areas show the crack generated during the drying process.

The synergy of reduced drying shrinkage, moisture loss, and percentage of humidity after the second and third tests cycle reveals a necessity of applying one cycle of wetting and drying treatment (Figure 5.5) of earth-based constitutive materials before application in the construction industry to improve the dimensional stability. This also reduces the moisture absorption capacity of the materials (Figure 5.7).

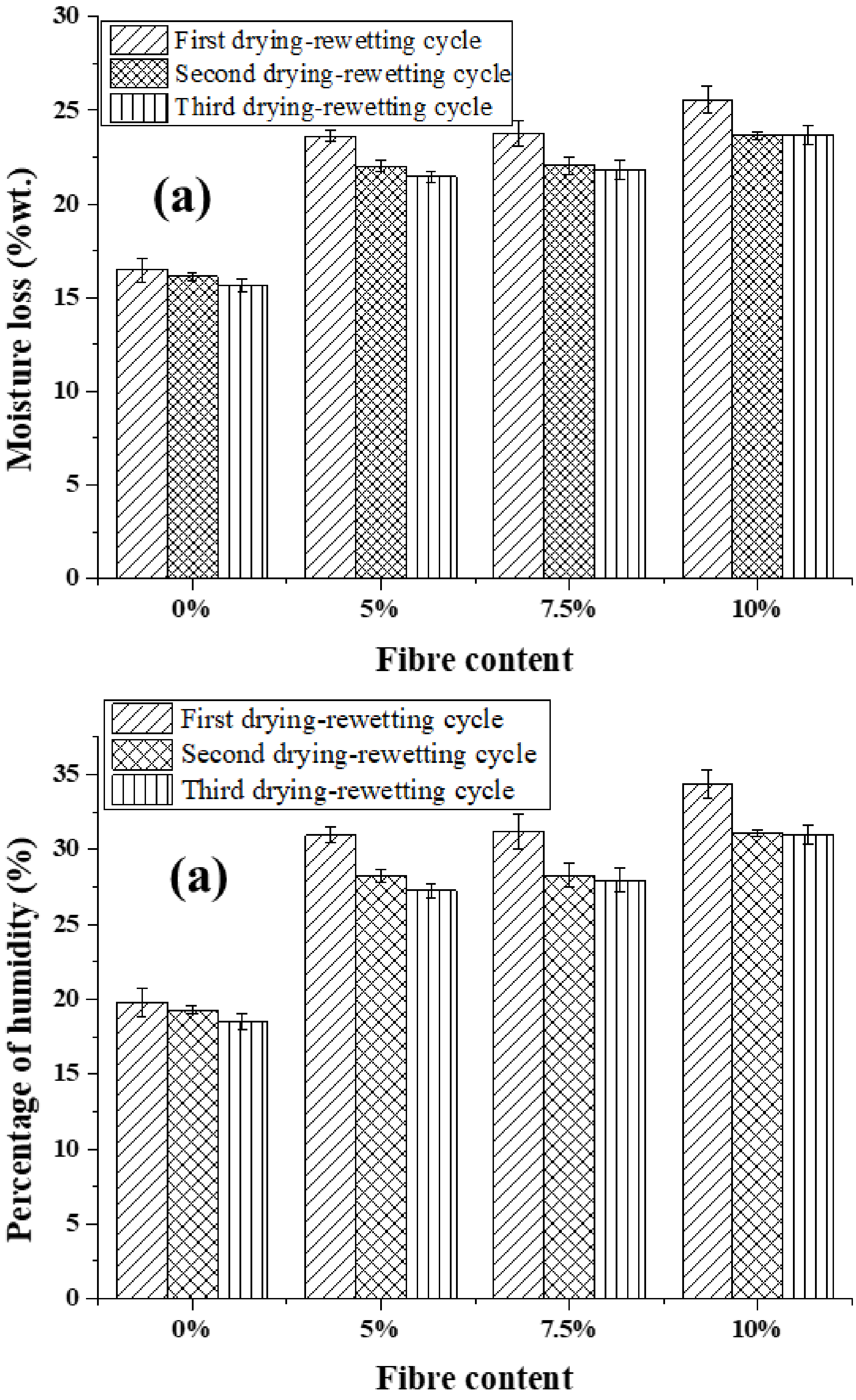

Figure 5. 7 Effect of drying following by rewetting cycles on the (a) moisture loss reduction and (b) percentage of humidity of earth-based materials

5.3.4. Strength and Fracture Toughness

The modulus of rupture (MOR) is directly attributed to the fibre/matrix bonding and the capacity of the reinforcement (fibre) (Correia et al., 2018). Thus it designate the ability of cellulose pulp to enhance the stress transfer when the specimen is under loading. While the fracture toughness is directly suitable to control the fracture or crack growth within the material (Mustapha et al., 2016), which prevents the catastrophic failure of the material. Table 5.4 shows that the composite reinforced with cellulose pulp presenting a higher MOR, SE and K_I compared to the unreinforced specimen. The MOE decreases with an increase in pulp content (except 10% carton pulp), whereas the SE and K_I increase with the pulp content. This indicates that the inclusion of cellulose pulp improves the hydration process due to its hydrophilic character, water absorption capacity, and porous structure (Figure 5.4b) absorbing the water during mixture which favoured the hydration reaction of the neighbouring paste.

The MOE of the reinforced matrix was lower than those of unreinforced composite, but their MOR which measures the specimen strength before rupture, was higher with 11 and 61 % increment compared to the composite reinforced with 10 %wt. of bamboo pulp and 5 % wt. of recycled waste carton pulp respectively. This shows that the pulps introduced some level of heterogeneity to the unreinforced matrix which eventually increased the overall strength and robustness of the composite. Even though there was a general increment in MOR initially, it was more visible with carton pulp which eventually dropped as the loading quantity increases (Table 5.4). This may be attributed to the fact that fibres tie each other when the amount is higher (Ismail & Yaacob, 2011) induce weaker fibre matrix bonding. For bamboo pulp reinforced composites, the initial increment was small of about 3% but gradually increases to 11% with the increase of as the pulp loading quantity (Table 5.4). The decrease in MOE with fibre reinforced indicates that there was a transition from a pure brittle unreinforced matrix to a composite along with ductility improvement.

Table 5. 4 Mechanical properties of cellulose pulp reinforced earth-based composites.

Type of fibre	Pulp content (%)	Modulus of rupture (MPa)	Modulus of elasticity (MPa)	Specific energy (kJ/m^2)	Fracture toughness, K_I (MPa.m$^{0.5}$)
Unreinforced	0	1.99 ± 0.18a	214 ± 93a	0.064 ± 0.018a	2.87 ± 0.18a
Bamboo pulp	5	2.04 ± 0.03a	150 ± 38a	0.24 ± 0.02b	3.76 ± 0.71ab
	7.5	2.15 ± 0.2a	83 ± 41.01a	0.31 ± 0.04bc	3.99 ± 0.32ab
	10	2.20 ± 0.04ab	80 ± 56.57a	0.36 ± 0.04cd	4.05 ± 0.42ab
Recycled waste carton pulp	5	3.21 ± 0.3b	122.5 ± 28.99a	0.33 ± 0.03c	4.52 ± 0.55b
	7.5	2.87 ± 0.3ab	89 ± 25.52a	0.36 ± 0.04cd	4.32 ± 0.24b
	10	2.91 ± 0.6ab	102 ± 38.97a	0.45 ± 0.06d	4.22 ± 0.48b

*Value with distinct letters in the identic column mean statistical difference resulted from Tukey post-hoc test ($p < 0.05$)

Specific energy (SE) is an important parameter for the fibre reinforced composite because it is closely related to the reinforcement efficiency of the fibre in the matrix (Ballesteros et al., 2019). There was a significant increase of about 416% in SE and 57% in fracture toughness with 5 %wt. reinforced with carton pulp (Table 5.4). A similar trend was experienced with bamboo pulp which had about 275% and 31% increment in SE and fracture toughness respectively, with 5 %wt. reinforcement. The inclusion of cellulose pulp in the matrix significantly improved the tendency to arrest crack propagation and toughness in the brittle matrix and thus prevent a catastrophic failure. Figures 5.8a and 5.8b present the crack profile of the underlying toughness mechanism of composite reinforced with bamboo pulp and recycled waste carton pulp. There is visible evidence of crack bridging with crack tip shielding in bamboo pulp composite (Figure 5.8a) and recycled carton pulp composite (Figure 5.8b). A similar crack bridging mechanism has been reported in the past on soil composite reinforced with bamboo fibre (Kolawole et al., 2017), straw fibre (Mustapha et al., 2016), and sisal fibre (Ojo et al., 2020). A maximum flexural strength and fracture toughness of 3.21 ± 0.3 MPa and 4.52 ± 0.55 MPa.m$^{0.5}$, respectively were obtained from composite with 5 %wt. of carton pulp. Kolawole et al. (Kolawole et al., 2017) reported from the study of cement stabilised earth-based block reinforced with bamboo fibre a flexural strength of 0.9 to 2.25 MPa and fracture toughness of 0.68 to 1.70 MPa.m$^{0.5}$. While Mustapha et al. (Mustapha et al., 2016) obtained a fracture toughness of 1.21 to 2.00 MPa.m$^{0.5}$. The higher value of fracture toughness of soil reinforced with pulp fibre compared to earth-based material reinforced with raw vegetal fibre may be related to the small size and high dispersion of cellulose pulp which enhance the resistance of crack propagation and expand the application of this building material where toughness and ductility are critically required.

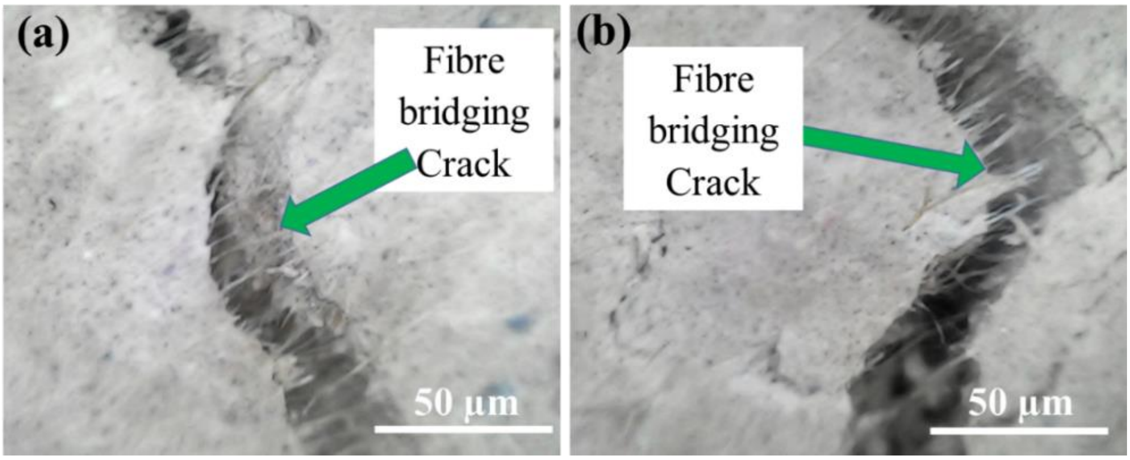

Figure 5. 8 Bridging of crack by (a) bamboo pulp and (b) recycled waste pulp during loading.

The improved synergy of MOR, SE and the fracture resistance of this composite suggests that the inclusion of bamboo cellulose pulp and recycled waste carton provides a robust earth wall material with higher ductility than traditional earth block.

5.3.5. Durability

Figure 5.9 presents the wearing percentage of earth-based composite reinforced with bamboo organosolv pulp and recycled carton pulp. It is visible from Figure 5.9 that the wearing percentage of cellulose pulp reinforced soil composite decreases with an increase in pulp fibre, except the formulation containing 10 %wt. of recycled carton pulp. The decrease in wearing percentage compared to unreinforced earth-based material was 18.53 %, 23.53 %, and 32.35 % respectively for 5, 7.5, and 10 %wt. bamboo pulp content. The decreasing value was 47.35 %, 48.82 % and 13.53 % respectively for 5, 7.5, and 10 %wt. for recycled carton pulp. This reduction implies that the inclusion of cellulose pulp in earth-based material improves wear resistance against external factors such as rainwater, wind, and any form of rubbing. Similar behaviour has been reported on soil brick composite reinforced with oil palm, bagasse and coconut fibres (Danso et al., 2015b), and cow dung (Manu, 2013). However, the higher value of wearing percentage of composite reinforced with 10 wt.% of carton pulp compared to 5 and 7.5 %wt. of carton pulp can be attributed to the high amount of pulp fibre that ties each other and induces lower bonding between pulp and matrix (Ismail & Yaacob, 2011). This is consistent with a lower fracture toughness of composite reinforced with 10 %wt. of carton pulp compared to 5 and 7.5 %wt. (Table 5.4). In addition, the wear value of all formulations remains significantly below the maximum allowable weight loss of 20 % established by the Indian Road Congress IRC: SP :89. (2010) for cement-stabilized earth materials during the wet-dry durability test.

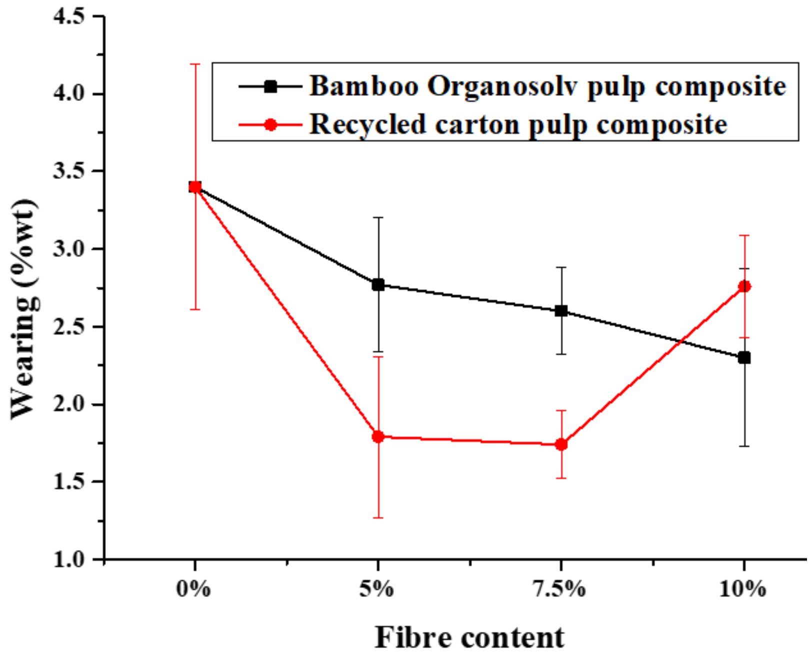

Figure 5. 9 Wearing performance of composite reinforced with bamboo organosolv pulp and recycled waste pulp.

5.4.Conclusion

This Chapter investigated the effect of cellulose pulp on flexural strength, fracture toughness, shrinkage cracking and durability of extruded earth-based composites. The conclusion from the study are:

- The inclusion of cellulose pulp in extruded earth-based matrix up to 5 %wt. significantly increased the drying shrinkage, water absorption, and apparent void volume, but reduced the shrinkage cracking and bulk density. This implies that cellulose pulp intercepts crack initiation during shrinkage and reduces the weight of the material.
- The synergy of reduced drying shrinkage, moisture loss, and percentage of humidity after the second and third drying-rewetting cycle reveals a necessity of applying one wetting and drying treatment of earth-based composite before the application in the

construction industry to improve the dimensional stability and reduce the moisture absorption capacity of the materials.

- As the amount of bamboo and recycled carton pulp increase, the specific energy significantly increases up to 463 % and 603 % with 10 % of bamboo and carton pulp respectively, while the modulus of elasticity decreases by about 63 % and 53 % for 10 %wt. of bamboo and carton pulp respectively. This indicates that the cellulose pulp induces the transition from a pure brittle unreinforced matrix to composite with improved ductility.
- Cellulose fibre inclusion significantly improved the flexural strength and the fracture toughness of the earth-based matrix by interception of the crack propagation. The predominant fracture mechanism observed on the reinforced composite was crack bridging.
- The wearing resistance of cellulose pulp reinforced composite is higher compared to unreinforced composite. This implies that the introduction of cellulose pulp in soil composite improved the durability of the material by reducing the degradation against rainwater, wind, and any form of abrasion.

The synergy of improved modulus of rupture, specific energy, fracture toughness, and durability of the produced composite reveals a more robust composite manufactured from bamboo and recycled carton pulp that presents a higher ductility than the traditional composites. This suggests that cellulose pulp composite can be adopted as a wall building material where strength, toughness, and durability constitute the critical requirement.

5.5.References

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CHAPTER SIX

6.0. Implication, conclusion and future work

6.1. Implications

The implication of this assessment of the feasibility of cellulose fibre reinforcement earth-based material for the design of a robust and environmentally friendly housing solution is

quite significant. First, the investigation of the pulping process of selected tropical plants suggests that the ambarella plant cannot be used as a source of cellulose production, while raffia fibre and cassava bagasse constitute a potential source of cellulose pulp and nanofibre. Unfortunately, raffia pulp and cassava bagasse pulp obtained by an organosolv process have a low aspect ratio (Table 3.2) and production efficiency (less than 25 %wt.)(Figure 3.1) compared to some non-wood resources such as the bamboo (production efficiency higher than 50%wt.) (Correia et al., 2015). However, an attractive combination of strength and toughness has been demonstrated for extruded earth-based material reinforced with cellulose fibre. This can be achieved by the reinforcement of cement stabilized extruded earth-based material with up to 7.5%wt. of bamboo organosolv pulp and 5%wt. of recycled waste carton pulp fibres. Above this, the modulus of rupture and limit of proportionality decrease, since fibre starts to tie each other (Ismail & Yaacob, 2011), ending in a decrease in cohesion in each matrix, and inducing weaker fibre-matrix composite.

Additionally, current work has shown improved thermal insulation properties and reduced density of earth-based matrices when reinforced with bamboo and recycled waste carton pulp fibre. This can be due to the insulation features of vegetal fibres as they have low thermal conductivity due to their porous structure (presence of lumen) (Robert et al., 2016). Therefore, this result suggests the application of this material to design earthen blocks for passive design building in order to solve both energy and environmental concerns.

Specifically, toughening of the cellulose fibre-reinforced earth-based composite was enhanced via crack bridging mechanism(Kolawole et al., 2017; Mustapha et al., 2016; Ojo et al., 2020; Savastano et al., 2009), precipitated by cellulose pulp fibre reinforcement. The result is consistent with the specific energy from the flexural test and suggesting the use of this fibre-reinforced composite in robust earth-based building material design for structural application where ductility is the main requirement.

Finally, it is interesting to note that the drying shrinkage, the moisture content, the percentage of humidity, and the water absorption of the composites increase with the increase in the content of cellulose pulp. Whereas the shrinkage cracks and wetting-drying durability decrease with the introduction of cellulose pulp fibre. This implies that the inclusion of cellulose pulp fibre in earth-based composite improves wear resistance against external factors such as rainwater, wind, and any form of rubbing. Further, the result shows a significant reduction in drying shrinkage, moisture content and percentage moisture after one wetting and drying cycle of the unreinforced and reinforced composite. This suggests applying a wetting and drying cycle of the composite before its application in the building industry to improve dimensional stability and moisture absorption. The behaviour of cellulose fibre as a water reservoir in an earth-based composite suggests the need to treat cellulose fibre before introduction into the soil matrix to reduce their absorption capacity and improve dimensional stability.

6.2. Conclusion

This work presents an experimental investigation of the potential application of selected tropical plants as a cellulose fibre source for reinforcement in earth-based block material and also assess the feasibility of soil matrix reinforced with two different cellulose fibres in order to promote the possible reinforcement of virgin pulp fibre with recycled pulp fibres. According to the results described above, the subsequent conclusion can be expressed.

- Due to the high value of wax (18.87%) and ash (16.05%) content, ambarella plant did not respond to soda and organosolv pulping.
- The organosolv pulping of raffia fibre and cassava bagasse appears more effective on the delignification than soda process, but the yields are lower (24%wt. for raffia and 12.3%wt. for cassava bagasse), compared to orgnosolv pulping of bamboo chips (55.6%wt.) at the same pulping condition. This results suggesting the high efficiency of bamboo source of organic solvent pulping.
- Inclusion of recycled waste carton pulp in earth-based materials significantly increases the moisture loss, the drying shrinkage, and behaves as a water reservoir for the composite. Thus there is a need to apply a treatment on the fibres to improve their dimensional stability and reduce their water retention capacity.
- The synergy of reduced drying shrinkage, moisture loss, and percentage of humidity after the second and third drying-rewetting cycle reveals a necessity of applying one wetting and drying treatment of earth-based composite before the application in the construction industry to improve the dimensional stability and reduce the moisture absorption capacity of the materials.
- By increasing the cellulosic fibre content, bulk density decreases, but water absorption and apparent void volume increase. This conclusion revealed that the transition area in the fibre–matrix becomes increasingly representative in the volume of the composite when the fibre content increase.
- As bamboo pulp content increases, the MOR and SE increase, while the MOE decreases during the bending solicitations. The optimum improvement in MOR was found to be

39% and 24% respectively at the dry and wet conditions at 7.5%wt fibre content in comparison to the control sample, and the optimum SE was achieved at 10 %wt. fibre content with an increase of 372 % and 977 % respectively at the dry and wet condition.

- The recycled waste carton pulp fibres insertion in soil composite improved the flexural strength of the composite by increasing MOR between 13 % and 56 % and SE between 623 % and 834 % in dry condition compared to unreinforced composite. While the saturated condition presents an increase in MOR of 40 % and SE of 2167 % at 5 %wt. of fibre content, which suddenly decreases with the addition of reinforcement.
- The minimum value of the modulus of rupture obtained from an unreinforced composite tested in the dry state was 1.6 MPa, which is within the typical range of values obtained in the literature (0.13-1.67 MPa) for unfired clay, suggesting the potential of the cellulose fibre reinforced earth material as well as an extrusion moulding mechanism for the production of earth composites.
- The introduction of cellulose pulp fibres yields a better thermal insulation behaviour of soil matrix, as at 7.5 %wt. recycled waste carton fibre content, k = 0.342 W/(m.K) which corresponding to 36.35 % of reduction compared to the control sample.
- The wearing resistance of cellulose pulp reinforced composite is higher compared to unreinforced composite. This implies that the introduction of cellulose pulp in soil composite improved the durability of the material by reducing the degradation against rain water, wind, and any form of abrasion.
- Earth-based composites reinforced by recycled waste carton fibres from the post-consumer waste present more advantages of resources and energy conservation and waste reduction as they can be used for construction and insulation. In addition, the successful replacement of virgin bamboo pulp fibres with recycled waste carton pulp fibres reduces the environmental footprint of the building material.

- Physical, mechanical and thermal behaviour of composite reinforced with 5 %wt. of recycled waste carton pulp and 7.5 %wt. of bamboo organosolv pulp fibre suggest a suitable utilization of these cellulose pulp fibres as a lightweight building material in all countries where there is a pressing need for a housing solutions to solve both energy and environmental concerns.

6.3. Future work

This study focus on the assessment of the reinforcement efficiency of cellulose fibre in extruded cement stabilised earth-based composite. According to the preliminary evaluation of the pH of the composite during the curing condition, the result shows that value of pH of the matrix slightly decreases with time due to the dissociation of the hydration products of cement (such as calcium hydroxide), which are responsible for the high alkalinity (pH > 11.62). According to Toledo et al. (Tolêdo et al., 2000), porous alkalinity medium (pH > 11), causes a progressive degradation of cellulose fibre and induces the reduction of its reinforcing performance. This result suggests the need to evaluate the stability and degradation of cellulose fibre over time. The degradation stage of cellulose fibre can link to the possible application of an accelerated carbonation process on the earth-based composite in order to reduce the pH of the composite and in consequence, raise the durability of the cellulose fibre.

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