

IMPLEMENTATION OF AGRICULTURE WASTES IN DIFFERENT CONSTRUCTION APPLICATIONS

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ABSTRACT

Implementation of Agriculture Wastes in Different Construction Applications

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Due to the growing population and increasing demand for more construction, much attention has been paid to environmental issues and the devastating effects of overgrowth in nature. Many challenges, including the 2030 challenge, have united developed countries to come together for a better and cleaner future. Canada is one of the allies in this challenge. In recent years, many alternatives to the main components of concrete have been introduced, which is known as one of the most widely used building materials. The use of waste in concrete as a substitute for main natural components (such as aggregates and sand) is one of the most popular methods to reduce environmental pollution by construction during these years. The use of tires, electronic components and agricultural waste are among the uses of waste as an alternative to concrete. Due to its ecofriendly natural, low cost and easy access, agricultural waste has received more attention than others. Several agricultural wastes such as hemp, coconut shells, and others were utilized successfully in producing agro-concrete. However, the limited availability of in-service data and stability of agro-concrete in the agricultural environment, which is very aggressive, is halting its acceptance in the construction industry. Therefore, this dissertation focused on examining the potential of using agro-waste in different construction applications. Effects of various factors including shape, replacement rate and physical properties of used agriculture wastes, type of binding materials, and exposure conditions on mechanical performance were evaluated. Also, a special type of concrete, known as Controlled low strength materials (CLSM), was tested as a potential hosting for high amounts of agro-wastes. Two types of CLSM were evaluated: a cement-based CLSM (i.e. with ordinary Portland cement) and zero-cement CLSM (i.e. with alkali-activated binder). Results showed the high potential of implementing agro-wastes in various construction applications, including agro-concrete, controlled low strength concrete for filling applications and zero-emission materials (i.e. zero cement). Moreover, alkali-activated CLSM showed a greater potential to incorporate a high amount of agro-wastes than that of cement-based CLSM. The research results represent a crucial point in getting these materials as acceptable as construction materials. Also, it will allow the agriculture industry to effectively recycle/reuse the

agro-waste, along with converting it to a valuable product. This will have a measurable impact on the Canadian specifications for concrete for farm and livestock buildings.

CO-AUTHORSHIP STATEMENT

Substantial parts of this thesis were either published in or submitted for publication to peer-reviewed technical journals and an international conference. All experimental work, data analysis, and writing of initial versions of all publications listed below were carried out by the candidate herself. The contribution of her research advisor and any other co-author, if applicable, consisted of either providing advice, and/or helping in the development of the final versions of publications:

1. **D. Ahadzadeh Ghanad**, A. Soliman, S. Godbout and J. Palacios “Properties Of Bio-Based Controlled Low Strength Materials,” Construction and Building Materials, (Manuscript # ID CONBUILDMAT-D-19-08664), **Accepted**, Included in Chapter 4
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LIST OF ABBREVIATIONS

AA	Alkali Activated
AAB	Alkali Activated Binder
AAM	Alkali Activated Mortars
CLCM	Controlled low strength material
FAO	United Nations Food and Agricultural Organization
OPC	Ordinary Portland Cement
C-S-H	Calcium Silicate Hydrate
PC	Portland Cement
Agro	Agricultural waste
SEM	Scanning Electron Microscopy

CHAPTER 1. INTRODUCTION

Since the beginning of human life, humans have always built shelters to protect themselves from external factors. The construction materials for these shelters has changed over time, from wood and stone to new modern residential buildings of concrete and metal [1]. The existence of concrete with its standard ingredients, cement, water, and stone dates back to several centuries [2].

As the construction industry grew, there was more need to manage the concrete sector better. Among several ideas, the production of a new generation of sustainable green concrete while maintaining the same strength and durability of the previous generation attracted more attention.

“Green concrete is defined as concrete which uses waste material as at least one of its components, or its production process does not lead to environmental destruction, or it has high performance and life cycle sustainability”[3]. One of the most important reasons for introducing a new generation of concrete to replace ordinary concrete is to curb environmental and social disruptions. According to earlier studies, concrete production has a significant impact on the environment due to its high carbon footprints, such as warming the earth and climate change[4]. Hence, the primary mission behind green concrete is to reduce the negative factors affecting climate change such as replacing cement, as one of the main leading to CO₂ production, by non-cement concrete (geopolymer or Alkali activated concrete) [5], using a sustainable alternative for fine or coarse aggregates such as usage of agricultural waste as a replacement of main components [6] and finally changing the main structure of concrete to address a new primary role to help the environment such as porous concrete which helps to save more ground water[7].

Meanwhile, population growth will lead to high demand for construction, which represents a challenge to our natural resources [8, 9]. Regarding the existing record, it is expected that by the end of the year 2100, the world's population would grow 11 billion[10], which will be accompanied by a massive demand in all industries. This demand will most likely be seen in the construction and food industry.

The annual production of more than 10 billion tons of concrete has made concrete one of the most popular and most essential buildings. This overuse has caused us to face the natural resource crisis.

In addition to the impact on the construction industry, this deficiency also expands the environment [2, 10, 11]. By 2050, it expected a dramatic increase up to 18-billion-ton in concrete production. This rate shows a considerable amount of natural resources need to produce concrete [12, 13]. The disturbing natural resource situation has left officials thinking of finding alternatives for these materials.

On the other hand, considering the growth of various industries, along with the production of products, a significant amount of waste production is also generated. A considerable amount of these wastes, such as agricultural wastes, can be used as alternatives to the main components of building materials[14, 15]. The volatile situation of the concrete industry makes it possible for the industry to use such wastes. Many successful results of the use of such wastes are available in the literature [12-20].

This study investigates the role of agricultural wastes in the construction industry. Chapter two reviews previous studies in this area. Chapter three discusses the properties and behaviour of these green concrete by investigating the behaviour of agro-wastes as coarse aggregate and fibres in concrete. The fourth chapter deals with the property's behaviour of agricultural waste as fine aggregate in a special type of concrete (i.e. Controlled low strength material). In the fifth chapter, green cement (i.e. alkali-activated materials) has been explored to make greener materials by removing cement in "agro-waste Controlled low strength material." Finally, the last chapter summarizes the main contributions and future research.

CHAPTER 2. LITERATURE REVIEW

Every year, the construction industry affects biodiversity by creating pollution such as land occupation, air pollution and excessive aggression to the environment. This issue makes researchers think about some new approaches to solve the influence on biodiversity by constructions and construction material. On the other hand, waste management, with the proposal to use agricultural wastes in building materials, has not only taken steps to help manage waste and reduce energy consumption and manage the financial sector but also using these wastes in building materials, has significantly reduced the amount of environmental pollution in the field of construction and construction materials production. Due to continued food needs, there is always an endless source of raw material by the Agriculture industry to produce these wastes for different industries such as the construction industry. In recent years, many researchers have investigated the use of these agricultural wastes in a variety of building applications. This study reviews the research conducted in this basin.

2.1 INTRODUCTION

The population grows in the last decade; address to unpredictable construction grows, which along with natural resources demand. Based on Maslow's pyramid of human needs, Physiological Needs such as food is in the head of the needs[16]. This need makes the agricultural industry one of the most functioning industries. The expansion in the agricultural industry created problems such as the management of agricultural waste[17, 18]. Animal waste such as manure, food processing waste, crop waste, and hazardous and toxic agricultural waste are different classes of agro-waste, which are in different forms (liquids, slurries, or solids). This agro-waste variety is one of the main reasons that waste management is facing difficulty[19, 20].

Agricultural industry classifies the world based on the type of Agriculture waste production produces and location into six groups: North America, South America, Europe, Asia, Africa, and Australia. For example, fig. 2.1 Shows North America from 1961 to 2014. [21, 22]. According to information published by FAO (United Nations Food and Agricultural Organization), although the amount produced varied for different products each year, it rose from 1961 to 2014. This increase in the production rate of the industry, coupled with a significant increase in the total agricultural

waste products, presents challenges for waste management[21, 22]. It is noted, however, that this growth rate is not equally divided in each of the six presented sectors. Each of these sectors, such as Asia, which alone accounts for about 4.4 billion tons in solid waste production[23], includes countries such as India, annually make 350 million tons of agricultural waste[20, 24-26].

On the other hand, this populace gain comes to have more construction needs. Increasing demand for concrete increases the need for main components such as sand and cement. Excessive extraction and production of these components cause irreparable damage to the environment. [27]. Hence along with all solutions waste managers comes with, such as burning, burial [28] despite their efficiency, are not responsive due to the excessive use of energy and financial resources[29]. Along with these solutions, the use of these wastes as an alternative to the main components of building materials, such as replacing agricultural residues in concrete instead of coarse and fine aggregates, has also provided many benefits, such as financial, energy, and environmental[20]. Landfilling and reducing the building material cost is another advantage of this replacement[20]. This study examines some of the roles that these agricultural wastes can play in the construction industry. These roles can be like replacing part of a building material's components, such as the role of fine aggregate for concrete. This study concludes by reviewing previous studies on the advantages and disadvantages of using waste.

2.2 DIFFERENT USE OF AGRICULTURAL WASTES IN THE CONSTRUCTION INDUSTRY

Table 2-1 shows some of the most widely used agricultural wastes in the building industry and the uses. As shown in this table, these wastes are used in a variety of modes and for different applications. In following are some of these applications. Some of these wastes, such as fly ash regarding worldwide production, does not have a primary resource.

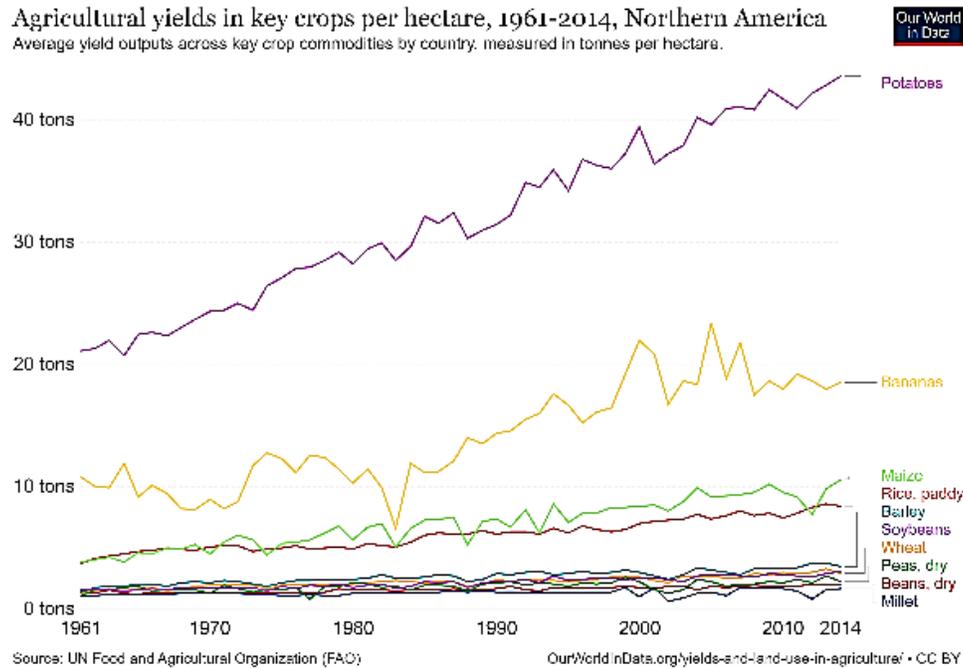


Figure 2.1 Agricultural yields across crop types [21, 22]

2.2.1 Insulation

Since buildings alone cannot control and create environmental conditions, they rely on the mechanical air conditioner. On the one hand, these air conditioners, besides being expensive, sometimes carry pollution such as noise and air pollution. Therefore, the use of thermal insulation is a great help in controlling and creating suitable conditions. The usage of these insulations varies from roof to floor or walls[20, 30]. As an example, Polystyrenes (PS) known as one of the materials used in daily life, especially the type of extruded polystyrene (XPS), which is one of the best-known insulators due to its productivity[31, 32]. Pinto et al. [33] showed in their study a significant microstructure and chemical composition similarity between the corn's cob and the extruded polystyrene (XPS). Pinto [33], in his work, produced microscopic images of this agronomic and polyester waste, which showed similarities in structure. The left image shows the image of crop waste on the scale of 400.0 μm , and the right image shows the polyester image on the scale of 500.0 μm . The results of these similarities attributed to the thermal insulation capability of these particles[20, 33]. Anabela et al.[34] estimated the thermal conductivity in his paper about $\lambda \approx 0.101 \text{ W/mK}$ for the corn cob [34].

Table 2-1. Different use of agricultural wastes in the construction industry

Type	Waste name	Uses	Location	Ref.
Solid	Newspaper	wood	Norway	[35]
Solid	Nappy roofing	tiles	-	[36]
Solid	Oil palm shell grains	Concrete	-	[37]
Solid	Plastic bags	Blocks, Concrete	-	[38, 39]
Solid	Freeze-dried blood	Bricks	British	[40]
Solid	Bottle	Bricks	Caribbean island	[41]
Solid	Smog	isolation	Bangkok	[42]
Solid	Mushroom	insulator Panels	-	[43]
Solid	unsorted plastic	asphalt	-	[44]
Solid	Rice Husk Ash (RHA)	Concrete	-	[14, 45]
Solid	Steel slag	clay bricks	China	[46]
Solid	Sludge	Concrete	-	[47]
Solid	Silica fume	Concrete	-	[48]
Solid	Fly Ash	Concrete	-	[49]
Solid	Inert	Concrete	-	[50]
Solid	Sewage sludge	Concrete	-	[51]
Solid	Oil sands drill cuttings	Concrete	-	[52]
Solid	Oil palm shell (OPS)	Concrete	Indonesia, Philippines	[53]
Solid	Coconut shell	Concrete	Asia and East Africa	[54, 55]
Solid	Corn cob	Concrete	USA	[33, 34, 56, 57]
Solid	Rice husk	Concrete	-	[58]
Solid	Tobacco waste	Concrete	India	[59, 60]
Liquid	Wastewater	Concrete	Singapore	[47]
Solid	Sugar cane bagasse ash	Concrete	India	[61]
Solid	Oil sand waste	Concrete	Canada	[62]
Solid	Palm oil clinker (POC)	Concrete		[63]

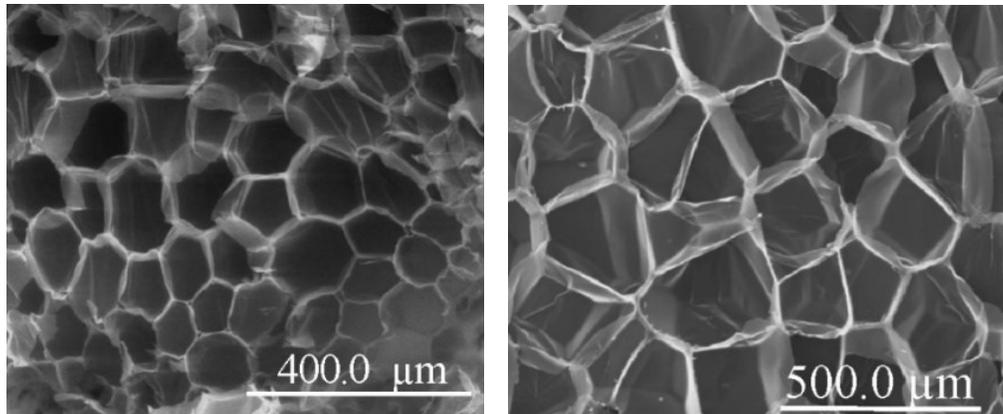


Figure 2.2 The left image shows the image of crop waste on the scale of 400.0 μm , and the right image shows the polyester image on the scale of 500.0 μm [33].

Flax is another agro-waste which has $\lambda \approx 0.4 - 0.5 \text{ W/mK}$ thermal conductivity coefficient[64].one of the primary usage of flax in the construction industry is a recommended isolation material in the areas without high static strains for the "breathing" buildings[64, 65]. Usually, the Flax shape depends on the usage of them. For instance, less ramified and taller size for fibres than oil format. About 75% of flat fibres by 8% ammonium Sulphate and 18% polyester fibres used as felt or plates insulation to be able about 30 kWh/m³ Thermal conductivity. Compares to other insulation materials, this material shows low energy realize [65]. Another agricultural industry with a historical background as the oldest agro-waste is hemp (figure 2.3). Hemp mostly suggested to be used as a floor, roof, and wall insulation regarding $\lambda \approx 0.040 \text{ W/m K}$ thermal conductivity[66].

Table 2-2 Agro-waste density and thermal conductivity as an insulator

Agro-waste	Density (kg/m ³)	Thermal conductivity (W/mK)	Ref.
Corn cob	300-330	0.097-0.101	[20, 30, 33]
Coconut coir	300-350	0.047-0.085	[20, 30]
Flax	311-336	0.4 - 0.5	[20, 30, 67]
Hemp	860	0.040	[20, 30, 68]
Bagasse	90-140	0.047-0.050	[20, 30, 69, 70]
Rice husk	150-175	0.046-0.056	[20, 30, 71]
Durian peel	400-875	0.065-0.150	[20, 30, 72]
Oil Palm Leaves	800-1000	0.118-0.240	[20, 30, 72]



Figure 2.3 Hemp in concrete.

2.2.2 Binder material

The use of cement as a staple material in the construction industry has been around for a long time, and its active role is undeniable. However, its deleterious effects on the gongs are also significant. Therefore, researchers have introduced many alternatives to cement in recent years.[73, 74]. Bagasse ash, as a cement replacement, is one of these alternatives. The residue, which was studied by Amin et al.[75] produced some exciting results. This sugar residue has been identified as a suitable mineral and pozzolan, which along with reducing 50% chloride emissions, maintaining the mechanical properties of hardened concrete at a replacement time of 20% cement ratio [75].

Also, Manasseh [76] reviewed some agro-waste ash such as groundnut husk (GHA), Acha husk (AHA), Bambara groundnut husk (BGHA), Bone powder (BPA), Groundnut husk (GHA), and Wood ash (WA) as a potential partial replacement of cement. Regarding his paper by increasing the percentages of replacement of cement by these ashes due to increasing the SiO_2 and decreasing CaO and C_3S generally address to a lower compressive strength than reference ones[76]. The different behaviours and properties shown in different studies indicate the different nature of these wastes — for example, the pozzolanic essence of GHA and RHA[76, 77]. Rice husk ash known as waste, which has a large amount of silicon carbide (SiC), silicon (Si), silica (SiO_2). This waste in concrete due to its pozzolanic structure can increase the durability of concrete in the long run along with proper efficiency and reduce permeability. It must be considered that the reactivity of RHA can affect the quality of the final concrete[78-81].

Among those who used RHA in concrete as a substitute for cement can be the article by Ramasamy and Biswa[82], Xu et al.[77] and Tashima et al.[83] Increased mechanical properties of concrete, increased strength, and reduced water absorption is among the advantages of this material. Xu et al.[77], in his paper, estimated the combustion temperature of this residue to be 600 ° C by performing an X-ray test to obtain the desired reactive material[77].

2.2.3 Reinforcement

Agricultural waste played a significant role in the construction industry. These roles differed in terms of the potential in agricultural waste, their contribution to other roles. One of these roles is the use of agro-waste in concrete to reinforced concrete. Brink and Rush[84] suggested a design procedure for regular reinforced concrete by steel to use bamboo instead to reinforce it [84]. After that, many researchers, including Kumar[85], have worked on this idea. Bamboo is one of these agricultural wastes that, due to its high properties and proportional appearance, has been able to play the role of steel for concrete in many places. In his study, Kumar[85] referred to bamboo as a “Green gold.” The reason for this naming can be the full range of uses of this plant. Also, due to its unique characteristics such as excellent flexibility and affordability and availability at the site of production, this agro-waste is introduced as a suitable alternative to reinforcement concrete

Figure 2.4. Eco-Friendly, low cost, higher bamboo stiffness than steel, are some of the reasons why bamboo used instead of steel. However, bamboo varies depending on several factors, such as soil conditions, age, climate. The use of these structures varies from low-cost construction such as low-cost buildings to eco-friendly structures such as urban furniture[85]. Also, Ghavami[86], in his paper, investigated the behaviour of reinforced lightweight concrete by bamboo (BRLC) beams and compared them with steel. Based on his results, bamboo is an excellent alternative for steel in this case, which is regarding the specific strength (SWR). Bamboo’s SWR is six times greater than steel, and Bamboo’s tensile strength can reach up to 370MPa [20, 86].



Figure 2.4 Bamboo as Reinforcement [85]

Kumar [85] also outlined in his study a comparison between concrete, steel, and bamboo carried out by Bhalla[87]. **Figure 2.5** shows this comparison. In this comparison, the ratio of tensile strength to density((SWR)) is superior to the other two.

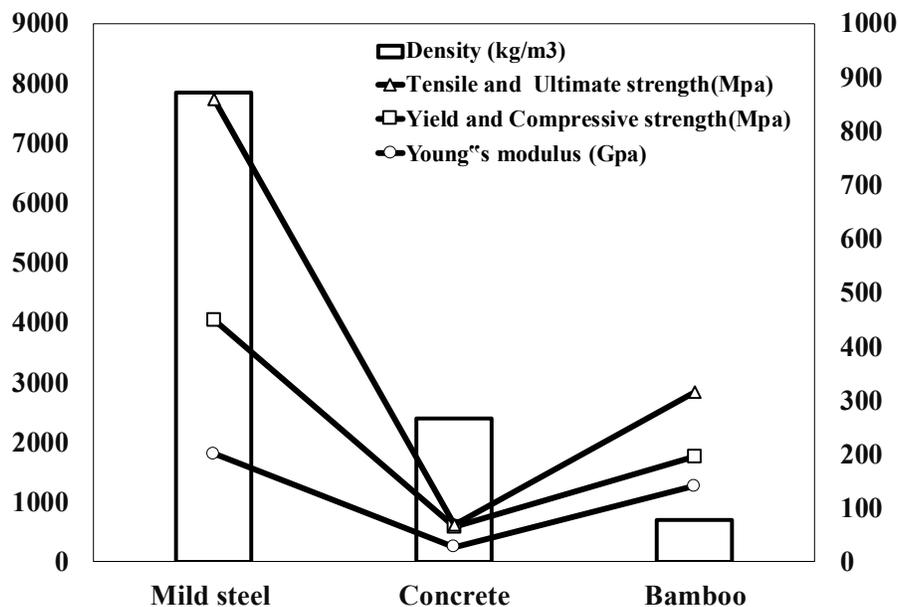


Figure 2.5: Comparison of properties between Mild Steel, Concrete and Bamboo [85, 87]

Although the use of bamboo had some brilliant results, however, due to the specific growth conditions of the environment, the researchers are considering the next option for steel replacement[88]. Including Priyadarshini Das[89], who studied rattan cane as an alternative to

steel in reinforced concrete. According to these results, the use of rattan cane, although not as brilliant as bamboo, was introduced as a viable alternative to steel for environmental reasons[89]According to many studies done by researchers, although rattan cane flexibility is higher, steel got better results in strength. According to that, using rattan cane as a reinforcement for concrete is better to be in nonstructural structures[89, 90].

2.2.4 Fibre reinforcement

The use of various fibres, such as steel and glass fibres, to reinforce concrete has yielded good results[91-94]. Hence, as with other cases, researchers have tried to introduce more eco-friendly and cheaper cases. Therefore, the use of various agro-wastes as fibres for reinforcing concrete was studied. These include sugar cane bagasse, coconut, corn stalk, and banana fibres [95, 96]. Joao[95], also in his study, compared three agro-waste as fibre in concrete with epoxy concrete. Figure2.6 Illustrates this comparison. Toughness and energy fracture both improve by adding coconut fibre and sugar cane bagasse. However, by adding banana fibres, just energy fracture improves and does not help the toughness of concrete as well as two other agro-wastes. Also, based on studies, sugar cane and banana fibre decrease the flexural properties compared to the polymer[91, 97]. Also, compared to other fibres such as glass and carbon, coconut fibre gives better results in epoxy polymer concrete[95].

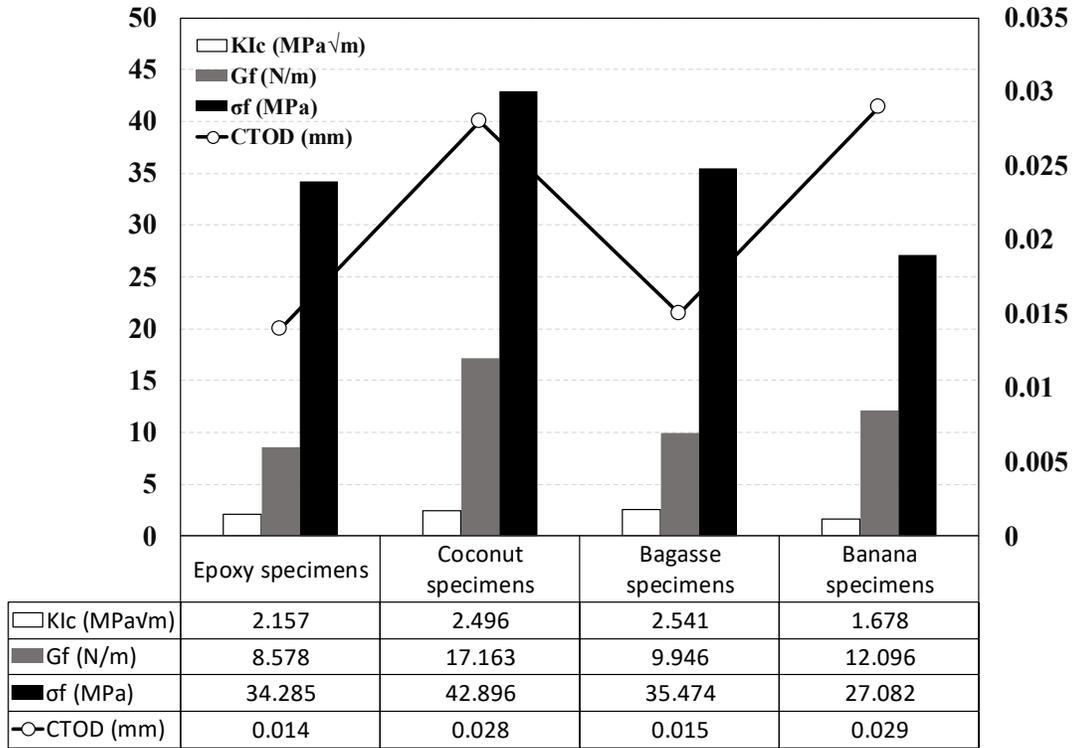


Figure 2.6 Comparison between bananas, bagasse, coconut agro-waste as fibre and epoxy concrete [after 94]

2.2.5 Aggregate

The use of agricultural waste as a replacement of materials in the building industry, depending on the apparent and mechanical properties of waste, has different uses. Regarding this state, the use of agricultural waste as aggregate in concrete is more investigated than other uses. This use varies depending on the age, type, and size of the aggregate can make differences in concrete properties[2, 20, 98-100]. On the other hand, the use of these agricultural wastes can be either raw or optimized. **Figure2.7** shows the three-stage rice husk provided by Raman[101]. As shown in the illustration, the use of this waste at each stage has different properties that make it suitable for use in the construction industry. This use can be either coarse or fine aggregate. Each of these alternatives comes with results that we will discuss separately from previous studies.



Figure 2.7 Rice husk from Selangor, Malaysia during the process[101, 102]

2.2.5.1 Coarse aggregate

Partially or complete replacement of coarse aggregate in concrete by agro-wastes such as oil palm shell[103](**Figure2.8**), coconut shell[104-106](**Figure2.9**), oyster shell[107](**Figure2.10**), periwinkle shell[108, 109](**Figure2.11**), Rubber seed shell[110](**Figure2.12**), Coconut shell[54, 105-107, 111-114] (**Figure2.13**) and Corn cob[56, 57, 113] (**Figure2.14**) were studied by several researchers. Mannan is among those who have studied utilized Oil Palm Shell (OPS) as a coarse aggregate and investigate its mechanical properties up to the age of 28days. The results showed that the lightweight concrete has a density of about 1850 kg/m³ and mechanical strength in the range of 20 and 24 N/mm², which can assume as coarse aggregate for lightweight structural concrete[115]. In the Ponnada studies, the replacement of only part of the coarse-aggregate concrete with Cockle Shell indicates a decrease in compressive strength. However, a combined fine and coarse aggregate replacement with different Cockle Shell sizes yielded better results. In his study, a 10% replacement of coarse aggregate by these waste gives about 28.4Mpa compressive strength in 28 days [116].

Replacement of coarse aggregate with coconut shell is associated with reduced compressive strength and density[106, 112, 117]. Kanojia[117] reported this in his study of a 22% reduction in compressive strength and 7.5% in density in 28 days with a 40% replacement. This reduction in density address to lighter construction[117]. The use of this waste in Faladeh et al. [109] research

shown periwinkle shell is not recommended to use in heat-resistant concrete structures. Also, the lower water-to-cement ratio in the same mixture yields a better compressive strength ratio.



Figure 2.8 OPS aggregates with and without fibres[103]



Figure 2.9 Cockleshell[118]



Figure 2.10 Oyster Shell[107]

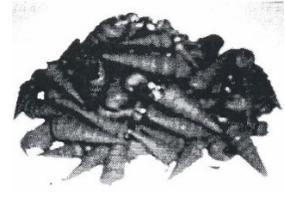


Figure 2.11 Periwinkle Shells[109]



Figure 2.12 Rubber seed shell (left side) and crushed rubber seed shell (right side)[110]



Figure 2.13 Coconut shell as Aggregates[114]



Figure 2.14 Granulate of corn cob[57]

2.2.5.2 Fine aggregate

In most studies, Agro-waste used as a replacement of sand (fine aggregate) in concrete as ash. Also, studies show that usage of agro-waste as a combination of fine and coarse aggregate shows a better result than alone [6, 119]. Loh et al.[120] introduces Bagasse as a fibrous waste of sugarcane, which is crushed and separated from juice[120]. The primary purpose of sugarcane bagasse (SCBA) is fuel in sugar industries. The problem starts from where the use is not complete, and the rest of this baggage is known as waste. These wastes become problematic when we consider sugarcane production as one of the leading products in the country, such as India. The use of these wastes in concrete as fine aggregate, especially in these areas, is a strong point in waste management and environmental protection[121, 122]. Based on Sales and Lima's studies [123], SEM analyses show Quartz in SCBA structure. Based on this finding, they recommended using this ash as a potential substitute for fine aggregate that presents high binding properties[123].

Cork is another agro-waste that can be an excellent replacement for sand regarding its high sound absorption, low thermal conductivity, and water resistance. Although the concrete that contains this material exhibit, a lower compressive strength has[124, 125]. Sawdust also reduces the compressive strength as well as cork, but the cost efficiency, which is about 56% lower than sand, makes it an excellent substitute for sand in nonstructural constructions such as pavements[126].

Decreased strength by adding these residues as a suitable substitute for fine aggregate led to the use of these residues in nonstructural structures. The use of agro-waste in controlled low-strength materials (CLSM) is one of the suitable alternatives for these wastes. In CLSM, bleeding, flowability, and compressive strength play a significant role[127-130]. Menya et al.[131] In their study used Treated oil sand waste in CLSM and find out that by adding this waste in CLSM, the bleeding decreased, and the flowability increased, which regarding the standards required redosing water contained to achieve requested demand. In his paper, the replacement by TOSW increased the compressive strength regarding reference one from 4.7 to 6.8 in 28 days.



Figure 2.15 Oyster shell[6]



Figure 2.16 Reed ash [6]



Figure 2.17 Cork [6]

2.3 Discussion

The increase in population rates in recent years has driven demand for more construction. On the other hand, according to the information available, the demand for the agricultural industry has also risen significantly. This sudden increase was unexpected and has accompanied by problems such as waste management, especially in the agricultural sector. The sudden rise has also accompanied by threats to natural resources such as sand, which have a significant impact on the construction industry. That is why using agricultural waste in the construction industry has been a way to solve multilateral problems[17, 132].

Based on the presented sections and results, we can see that the range of use of agricultural waste in the construction industry is vast, depending on the individual properties of each waste. This use can apply to insulators. XPS is one of the most popular insulators used in buildings, especially on walls, floors, and ceilings, due to the many similarities in appearance to the building, it can replace by corn cob. According to ASTM[133], thermal conductivity is the amount of heat that material can pass in the unite of time and thickness and at a specific time. Proper insulation is inversely related to thermal conductivity. The lower the thermal conductivity, insulation material works better for controlling space temperature. According to this standard, **Table2-2** shows different agro-waste and their thermal conductivity, which hemp shows a better result[20, 134]. Although some agro-waste in different roles, such as reinforced fibres, does not have enough strength regarding other agro-waste fibres such as Banana fibres, they have the right energy fissure that can play an insulation role as well[91]. Using agro-waste in concrete, along with being optimized, may cause problems such as low durability, especially in alkaline environments. In terms of strength and insulation, although these residues sometimes appeared weaker or stronger than non-residual concrete, they were associated with a standard framework. However, despite the problems that may arise later in the concrete durability process, the use of these wastes is more desirable[20].

On the other hand, in many cases, the high cost of steel in production and operation makes it more attractive to use a replacement such as bamboo, which has both compressive strength(41.02 MPa)and excellent tensile strength (370MPa). Availability, low cost, friendliness to the bamboo structure are other reasons for using this material in place of metal, especially in low strength structures. [84, 88, 107, 135, 136]. Also smaller in size than bamboo is the agricultural waste used as aggregates in concrete. This use has particularly useful when applied to agricultural waste in both coarse and fine aggregate sizes[8, 20, 37, 123]. Another area where the use of agro-waste is associated with good results, and sometimes its use is better than the control sample is in CLSM. In this group, as long as the results of durability and hardened are of value, fresh tests should be standardized. Which in this case, Agro-waste shows an excellent result spacially in reducing bleeding and increasing flowability[127, 131, 137-142].

2.4. The waste preparation process for use in the construction industry

As seen in the above categories for the use of agricultural waste in the construction industry, agricultural waste needs to be prepared to be usable in this construction industry. This preparation can have different shapes depending on its type and size. Although these processes may be costly on a small scale, they are economical on a larger scale by reducing greenhouse gases, preserving environmental resources for future generations, and replacing these materials with long-term concrete substitutes[14, 143-147].

Not only are these processes different, but in the same process, different parameters such as temperature or size of inputs can be produced to produce a different waste output depending on the type of need in the construction industry[14]. Although this diversity creates more opportunities, it is not always mean perfection. Rice, for example, is one of the essential products in the agricultural industry (especially in the Middle East), and its waste has received considerable attention due to its due source of pozzolanic materials .Up to 75% of the ash mass-produced by burning rice husks is silica, which can play a major role in pozzolanic reactions. This use was first patented in 1924 by Pitt, which regarding the high amount of pozzolanic nature, was initially able to take a major step in the use of agricultural waste in the construction industry. [148-150]. For example, in the same sample of rice husk ash, several devices were made to control this amount and optimize it. Unburned carbon is one of the threats that can threaten as an example. In the prototype built from 1924 to 1974, this process took place without combustion and temperature control.[14, 151-154] Although this is a significant amount, it should be noted that this amount may be subject to changes in the final product structure during different preparation operations. As an example, it can be motioned that Ramezianpour et al.[14], have examined different types of rice husk ash during different processes to get to this point. **Figure 2.18** shows five different types of rice husk ash in one method but with different parameters [14].

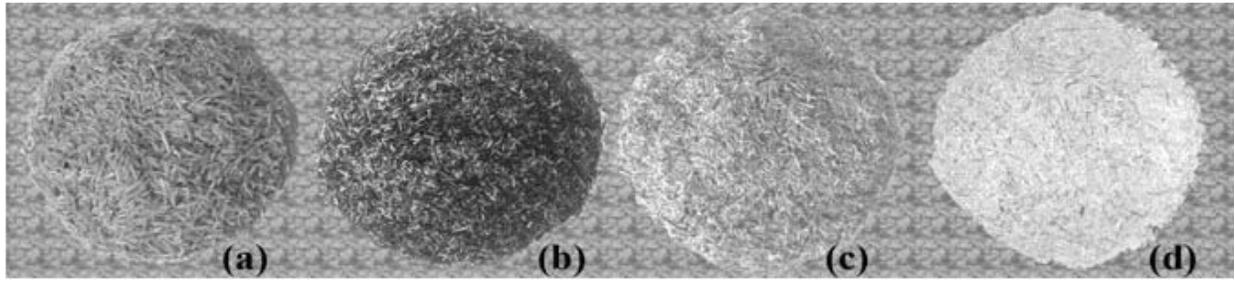


Figure 2.18 (a) Rice husk, (b) Rice Husk Ash with high carbon amount, (c) optimum Rice Husk Ash, (d) Rice Husk Ash with crystalline silica [14]

It should also be noted that in addition to the process of preparing these wastes, their birthplaces can also be different. For example, in **Figure 2.19**, Sankar et al.[81] showed in his article that rice has a different appearance with different main nature, which affects not only its external appearance but also its structure. Therefore, the type of ash and its main nature can also affect the durability of our final concrete product. In addition to all the points mentioned during the process of agricultural waste, it should be noted that control and accurate operation are the main points of this process. In uncontrolled processes, we can encounter a significant amount of impurities, which can affect the hydration process. For example, in the same sample of rice husk ash, several devices were made to control this amount and optimize it.



Figure 2.19 Rice from different locations and type in the process of preparation[81]

2.4 Conclusion

A review of previous researches yields the following points:

1. The use of agricultural waste in the construction industry has an economical benefit as a way for waste management.
2. Agro-waste can be used in various industrial applications, depending on their appearance, such as size, shape, and type, as well as mechanical properties.
3. The use of agro-waste as an insulator has associated with excellent results; especially the as heat and sound transmission insulators.
4. Bamboo's tensile strength can reach up to 370 MPa that present an excellent behavior as steel substitute in concrete.
5. About 75% of the concrete volume is coarse and fine aggregate, which increase the potential for implementation of agro-waste as a replacement for aggregates.
6. To achieve a better result, a combination fine and coarse agro-waste is recommended.
7. Increased strength and reduced water absorption are the most benefits of using agro-waste products in the concrete binder.
8. Ash agro-waste is considerable combined with binders.

The use of agricultural waste is a two-way innovation in the construction and waste management industry. Most of the waste used in the industry is mostly used to dispose of these wastes and has investigated by a process. Future studies will focus more on the properties and mechanisms of the materials made from these wastes as raw materials. This will save finance and time by eliminating the treatment period for these wastes. On the other hand, these treatment processes, such as incineration and ashes, are also associated with environmental pollution.

CHAPTER 3. USING AGRICULTURAL WASTES AS CONCRETE AGGREGATE

A dramatic annual increase in the population grows Leads to a climactic demand for concrete as one of the most trendy materials in the construction industry. This unforeseen increase has led to the overuse of materials such as cement and sand, which is one of the factors affecting damage. It should also consider that this dramatic increase in population, along with an addition in need for more construction, Leads to an expansion in need for a feed and a continuing rise in the demand for the agricultural industry as a significant source of food and apparel. The growth of the agricultural sector, which encompasses a wide range of fields, such as agriculture, harvesting fish and animal husbandry, has led to a significant increase in waste production as the crop has grown. Due to unforeseen impacts on demand and production, waste management and the environment are affected. This sudden rise in industries, made the scientists propose different solutions to decrease or remove the effects on the environment and along with our life. One of the suggested ways is to use agricultural waste in construction materials. Concrete, as one of the most widely used building materials in recent decades, has also been affected by this proposal. In this study, four different agricultural wastes used in concrete and fresh and hardened properties of concrete, with different percentages of waste, were investigated at different ages.

3.1 Introduction

3.1.1 Research Contributions

The primary objective of this research is to develop a new sustainable construction material/product incorporating agro-waste materials (green construction material) with adequate mechanical and durability performance for different construction applications, and especially farm buildings. This research is anticipated to help in reducing the amount of agro-waste materials

through in-situ recycling in new farm buildings leading to society, economy, and environmental benefits. The following is a summary for the main achievements, completed and ongoing research:

1. Various agro-waste materials were characterized and examined as a potential replacement for natural aggregates.
2. Agro-concrete mixtures with tailored engineering properties were designed to accommodate high percentages of each selected agro-waste materials as a total/partial replacement for natural aggregates;
3. Fresh and hardened properties for optimized agro-concrete mixtures were evaluated and compared to different construction specifications;

3.2 Background and Potential

3.2.1 Problem Definition

With more than 10 billion tons produced annually, concrete is an essential construction material [155]. It has predicted that the world's population will increase to 11 billion by the end of the century, which considerably increases the demand for concrete. It expected that concrete production would grow to approximately 18 billion tons by 2050 [156]. Extensive range use of concrete in constructional applications including bridge, roads, tunnels, and various buildings. For farm and livestock buildings, concrete is a strong competitor to other building materials due to its strength and durability in such a harsh environment. The high resistance of concrete to different aggressive materials, such as wastes generated by pigs, cattle, and poultry, makes it a good choice material for such structures. Also, the texture of the concrete makes smooth surfaces easy to clean. Concrete walls have a high resistance for fire, which will increase the safety aspects of various farm buildings. Many advantages are associated with the use of concrete in farm buildings.

However, the concrete industry is facing significant challenges concerning its ecological and environmental impact. The concrete industry consumes a significant amount of energy, water, aggregate, fillers, and other natural resources to produce cement and concrete. Moreover, at the end of its life cycle, construction waste from the demolition of concrete structures is another environmental impact [8, 157, 158]. Hence, there is some need to make this critical construction material compatible with the environmental requirements of the modern sustainable construction industry.

One viable way to increase the concrete industry's sustainability is to utilize industrial and agricultural wastes. Industrial waste materials, such as silica fume, fly ash, ground granulated blast furnace slag, and others, have been successfully used in concrete for a long time [8, 155-159]. Recently, agricultural solid wastes (i.e., agro-waste), such as oil palm shell, coconut shell, corn cob, pistachio shell, have attracted researchers as a replacement for natural aggregate in structural and non-structural concrete [108, 160-162]. The use of these agro-wastes as total or partial replacement of natural aggregates, which makes up about 60–80% of the volume of concrete [163], represents substantial energy saving, conservation of natural resources, and a reduction in the cost of construction materials. Also, it solves the disposal problem of agro-wastes helping in protecting the environment [164, 165].

This chapter will help in extending the knowledge about the performance of concrete incorporating agro-waste materials. Also, it will highlight the effects of variations in agro-wastes properties on the overall performance in specific applications. Moreover, it will initiate an investigation of the effects of the agriculture environment, which is considered a very aggressive environment for agro-concrete. This sum up will provide an idea about the durability of agro-waste construction materials, which is a significant concern for the agriculture sector [166, 167]. Recognition of these materials, and implementing in concrete by engineers, would pave the way for a wider acceptance of such sustainable construction materials.

3.2.2 Potential Applications for Agro-waste Materials in Farm Facilities

The agricultural wastes have the availability to use as a partial replacement for fine and coarse aggregate in farm concrete. Different concrete structures can found in farms reviewed in the following section, along with highlighting successful implementation for agro-waste in concrete.

3.2.2.1 Paving

Slabs on grade are the most common farm application for concrete. It used for feeding floors, feedlots, and floors in livestock buildings. The thickness of these pavements varies from about 10 to 20 cm. Livestock building floors and outside paved areas on which loads limited to livestock, cars, small trucks, and smaller farm equipment need to be only 10 cm thick. Areas subjected to heavier equipment, such as large trucks, grain wagons, and manure tanks may need to be as much as 20 cm thick [168, 169].

3.2.2.2 Concrete Walls

There are two types of “concrete walls”: insulated and non-insulated concrete walls. Agro-waste materials were used to produce a special concrete known as hemp concrete. It produced by mixing fibre hemp, lime, and water. Once it dry, it turns into a solid material that is stronger and harder than cement while weighing only one-sixth of the normal cementitious materials weight. Hemp concrete is more flexible than conventional concrete, giving it an advantage over conventional building materials, especially in areas prone to earthquakes. Hemp concrete [170]used in several applications, including the use as a plastering material for filling half-timbered constructions or for walls with a wood structure. Also, it is used for the production of insulating screeds on light or large floors and insulation for inclined roofs, through increasing the thermal inertia for the structures[170-172].

3.2.2.3 Fibre-reinforced concrete with vegetable fibre

Vegetable fibres contain cellulose, a natural polymer, as the primary reinforcement materials. The chains of cellulose form microfibrils, which are held together by amorphous hemicelluloses and lignin forming fibrils. The fibrils assembled in various layers to build-up the structure of the fibre. Fibre or cells are cemented together in the plan by lignin, which can be dissolved by the alkalinity of the cement matrix[173]. Ashour et al. [174].recommended the use of wood shavings and barley straw as fibre in the concrete. Results indicated that the thermal conductivity of materials decreases with increasing straw fibre content and increasing sand content.

3.2.2.4 Slatted floor

In livestock buildings, some parts of the pavement consist of a series of non-joined beams laid above a pit or a slurry channel. This particular pavement is called "slatted floor," and the structural elements are called "gratings" (**Figure 3.1**). Therefore, using agricultural waste as a partial replacement of fine or coarse aggregates in concrete used for the manufacture of slatted floors is a potential application that requires a high durability performance.

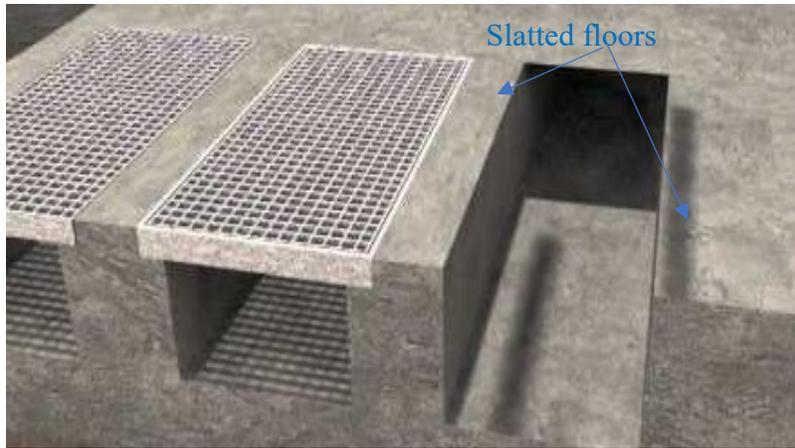


Figure 3.1 Slatted floors structure in farms

3.3 EXPERIMENTAL PROCEDURE AND SPECIMENS PREPARATION

The experimental work has divided into four main phases in this chapter only. The initial phase focused on characterizing different types of agro-waste materials. This experiment included visual examination and identifying physical properties such as moisture content, water absorption capacity, and stability in alkali environments. Moisture content and water absorption were evaluated for each received samples according to ASTM D4442 “Standard Test Methods for Direct Moisture Content Measurement of Wood and Wood-Based Materials” and ASTM C128 “Standard Test Method for Relative Density (Specific Gravity) and Absorption of Fine Aggregate,” respectively. The other three phases will cover experimental works on different types of construction materials incorporating agro-waste materials.

3.3.1 Agro-Waste Characterizations

3.3.1.1 Visual Inspection

Figure 3.2 shows the four coarse samples of agro-waste materials used in this study. The first sample (**Figure 3.2 a**) was labelled “**Topinambour(English name: Jerusalem artichoke).**” It was a very lightweight and looked like chopped corn stover waste. It had a light-dark brown colour. The second sample shown in **Figure 3.2 b** was labelled “**Saule and wood.**” It was a mix of wood waste with willow and had a brown colour and a smooth and shiny surface. Moreover, it was slightly heavier than the type “Topinambour.” The third sample shown in **Figure 3.2 c** was labelled

“**Saule.**” It looked like wood chips waste with variable sizes. The fourth one, shown in **Figure 3.2 d**, was labelled “**Miscanthus,**” which is an invasive grass. It looked like a wheat straw waste. It was a fibrous material and very lightweight. The particle size varies significantly within each sample. **Table 3-1** summarizes the range of sizes in each sample. It should mention that each type of agro-waste coded to make it easier.

Table 3-1 Particle size for as received samples

Samples	Topinambour (AWM1)	Saule and Wood (AWM2)	Saule (AWM3)	Miscanthus (AWM4)
Size	1.2 to 1.5 cm	1.0 to 2.5 cm	1.5-3.1 cm	0.01 to 3 cm



Figure 3.2 Different agro-waste materials: a) Topinambour (Code: AWM1); b) b. Saule and wood (Code: AWM2); c) Saule (Code: AWM3) and d) Miscanthus (Code: AWM4)

3.3.2 Physical Tests

Figures 3.3-3.4 summarize results for density, initial moisture content, and water absorption evaluated on “as received” samples for the four coarse wastes. It seems that all agro-waste materials had a similar initial moisture content (around 10%), except Saule and Wood (AWM2). AWM2 showed a low moisture content of around 1.6%. This amount may be attributed to its very dense surface, which probably had affected moisture exchange. This is in agreement with the density results reported in Figure 3.4. The densities for all agro-waste varied slightly, except for AWM2, which showed a significantly higher value compared with others (approximately the double). This confirms the high compaction for this type of agro-waste material, which can be ascribed to its production process—results for absorption rates for all agro-waste materials illustrated in Figure 3.5. Agro-waste materials (i.e., AWM1 and AWM3) showed a lower water absorption, which can be attributed to their lower surface area compared to other wastes. One

interesting point, the very dense agro-waste material AMW2 expanded once comes in contact with water showing a very high water absorption potential.

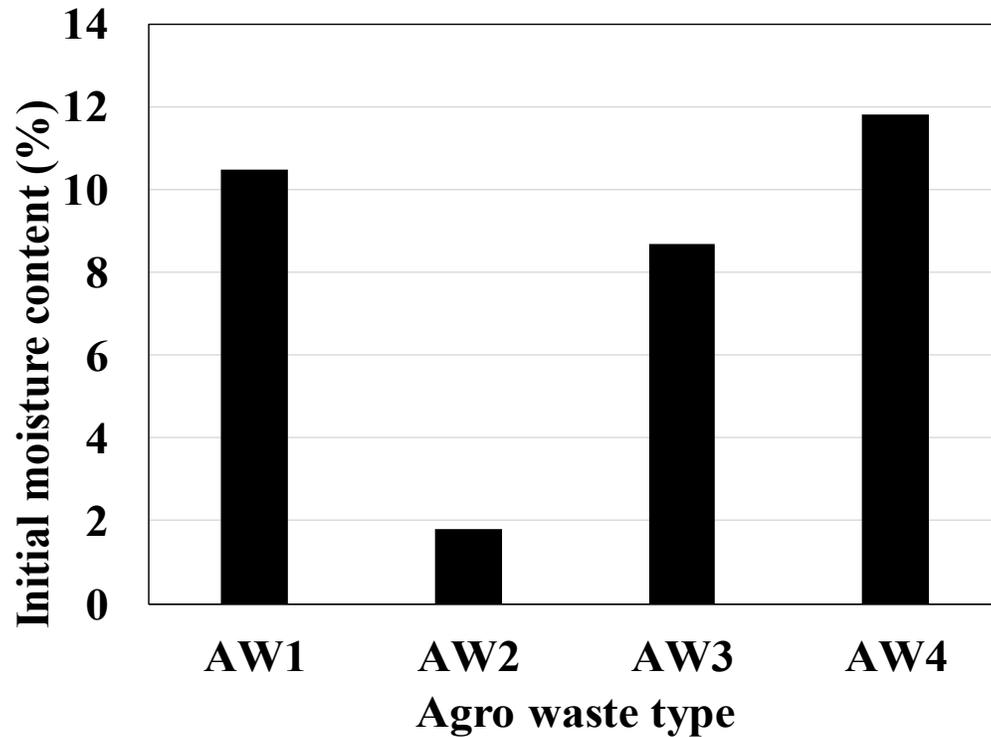


Figure 3.3 Moisture contents for as received samples of different agro-waste materials

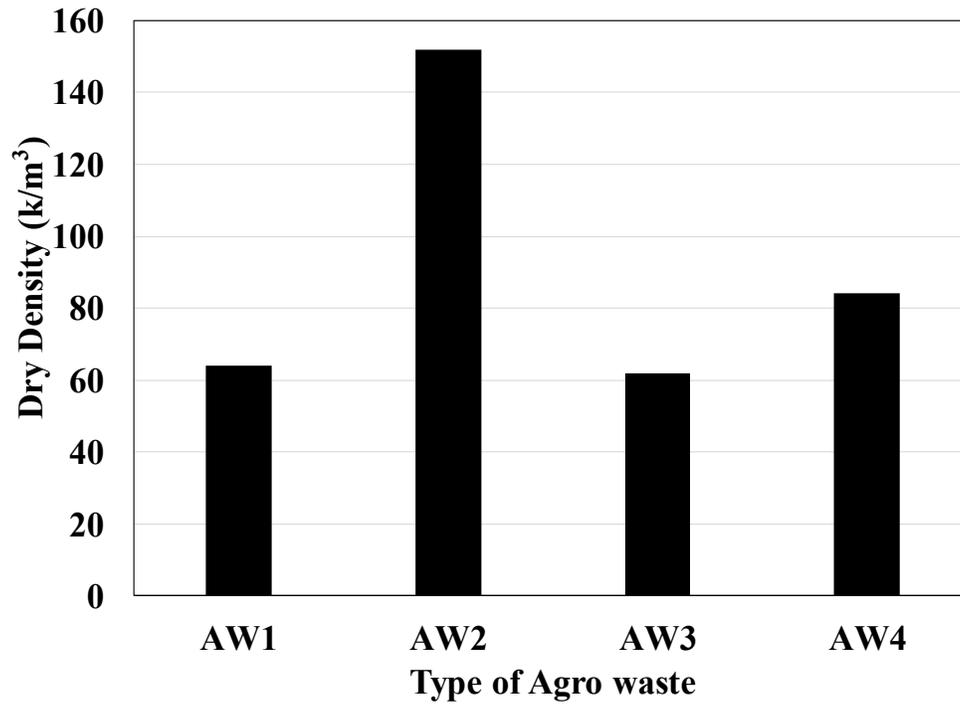


Figure 3.4 Densities for as received samples of different agro-waste materials

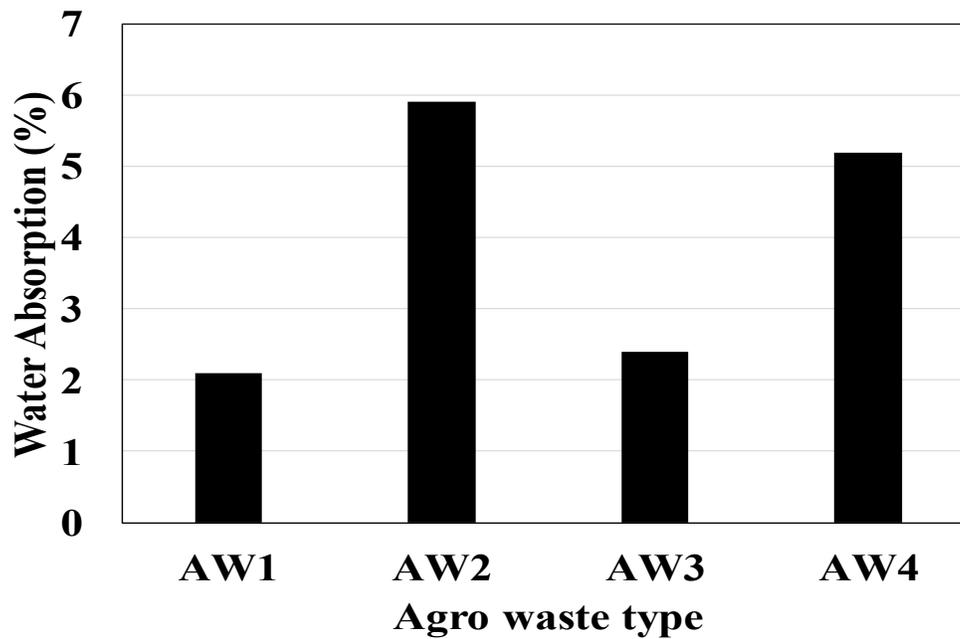


Figure 3.5 Water absorption values as received samples of different agro-waste materials.

3.3.2.1 Particle Size Distribution

Figure 3.6 shows particle size distributions of different agro-waste materials. It can be seen that agro-waste materials sizes vary between 5 to 25 mm. The medium-sized particles represent about 65 to 70% of the total particles in the range (1-3 mm). These particles at the medium size were flaky and angular. Bigger particles were parabolic with convex and concave surfaces and represented about 15 to 20%. The smaller particles, in the range of 80 μm to 0.5 mm, represent around 1 to 7%, and these particles mostly rounded and angular.

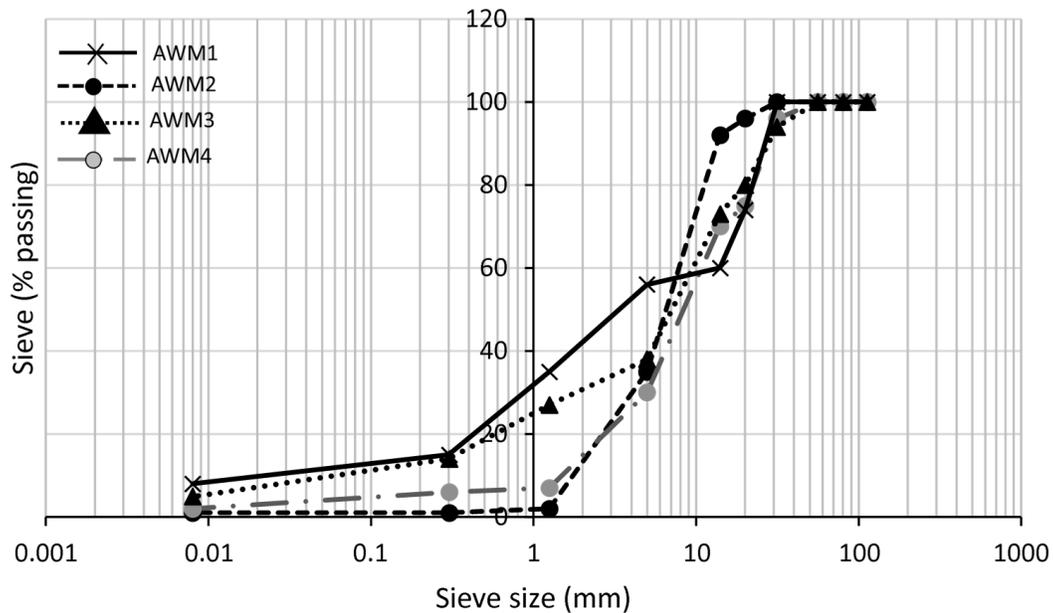


Figure 3.6 Distribution size for different agro-waste materials.

3.3.2.2 Microstructure Analysis

Figure 3.7 shows the microstructure for different agro-waste materials examined by Scanning electron microscope (SEM). The high void structure for AWM1 and AWM3 explain the lightweights for these types of agro-waste materials, as shown in **Figure 3.7a**. **Figure 3.7b** shows the microstructure for AMW4, which has a very high surface area in the fibrous form.

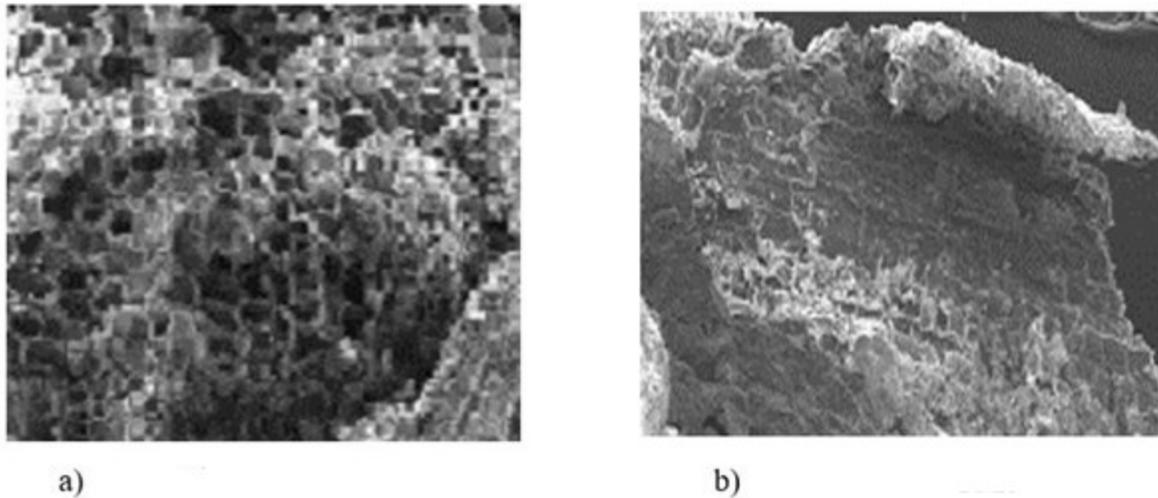


Figure 3.7 SEM image for selected samples from the used agro-waste materials. a) High void in AWM1 and AWM3 b) fibrous structure for AWM4

3.3.2.3 Stability in High Alkalinity Environment

A non-standard test was conducted on different agro-wastes to explore their resistance to alkali environments. The particles were stored for 24 hrs. in sodium hydroxide solution with a pH= 13. The sodium hydroxide selected as it commonly used as a treatment method for agriculture materials before using it in concrete. **Figure 3.8** shows the samples before and after the addition to the alkali solution.

The change in the solution color indicates the dissolution of organic materials. For AWM1 and AWM4, the waste becomes softer and a little sticky while maintaining the same features, including shape and size. For AWM2, the waste had absorbed all the solutions and increased in size significantly. In conclusion, AWM1, AWM4. Particular dry applications will be more suitable for AWM2.



Figure 3.8 Agro-waste materials before and after submerging in alkali solution.

3.3.3 Production of Agro-Concrete

This phase is dedicated to examining the potential of producing agro concrete using the supplied agro-waste materials as a replacement for natural aggregate. Different concrete mixtures were designed to incorporate various contents of agro-waste materials. Effects of adding these agro-waste materials on fresh and hardened properties evaluated.

3.3.3.1 Experimental Work

3.3.3.1.1 Materials

General Used (GU) hydraulic cement, according to the CSA-3001-03, was used as the binding material. **Table3-2** shows the chemical composition for the cement. The fine aggregate was natural riverside sand with a fineness modulus of 2.70, according to ASTM C136 (2014), specific gravity and water absorption of 2.51 and 2.73% determined by ASTM C 128 (2015), respectively. The coarse aggregate was siliceous/calcareous aggregates with a maximum size of 20 mm (3/4 inch),

the specific gravity of 2.697 kg/m³, and water absorption of 0.6%. Sieve analysis for both fine and coarse aggregates meet ASTM C33 (2018), as illustrated in **Figure 3.9**. A high range of water reducer (HRWR) in the range of 2.0% to 3.5% of the cement weight, and air-entrained in the range of 35–65 ml/100 kg of cement were also used to adjust the concrete flowability and air content. Tap water was used in all mixtures. Three types of agro-wastes added as a replacement of aggregate. It should be mentioned that based on Phase I results, AWM1, AWM2, and AWM3 selected to use as a replacement for natural aggregate with different levels. Due to its fibrous shape, AWM4 was used as an additive to reinforce the concrete (i.e., similar to the way of adding other synthetic fibres).

Table 3-2 Chemical and physical properties of cement

		OPC ⁽¹⁾
SiO ₂	(%)	19.80
Al ₂ O ₃	(%)	4.90
CaO	(%)	62.30
Fe ₂ O ₃	(%)	2.30
SO ₃	(%)	3.70
Na ₂ O	(%)	0.34
MgO	(%)	2.80
C ₃ S	(%)	57.00
C ₂ S	(%)	14.00
C ₃ A	(%)	9.00
C ₄ AF	(%)	7.00
Na ₂ O _{eq}	(%)	0.87
Loss on ignition	(%)	1.90
Specific gravity	--	3.15

(1) GU cement produced by Lafarge cement plant, Factory at St. Constant

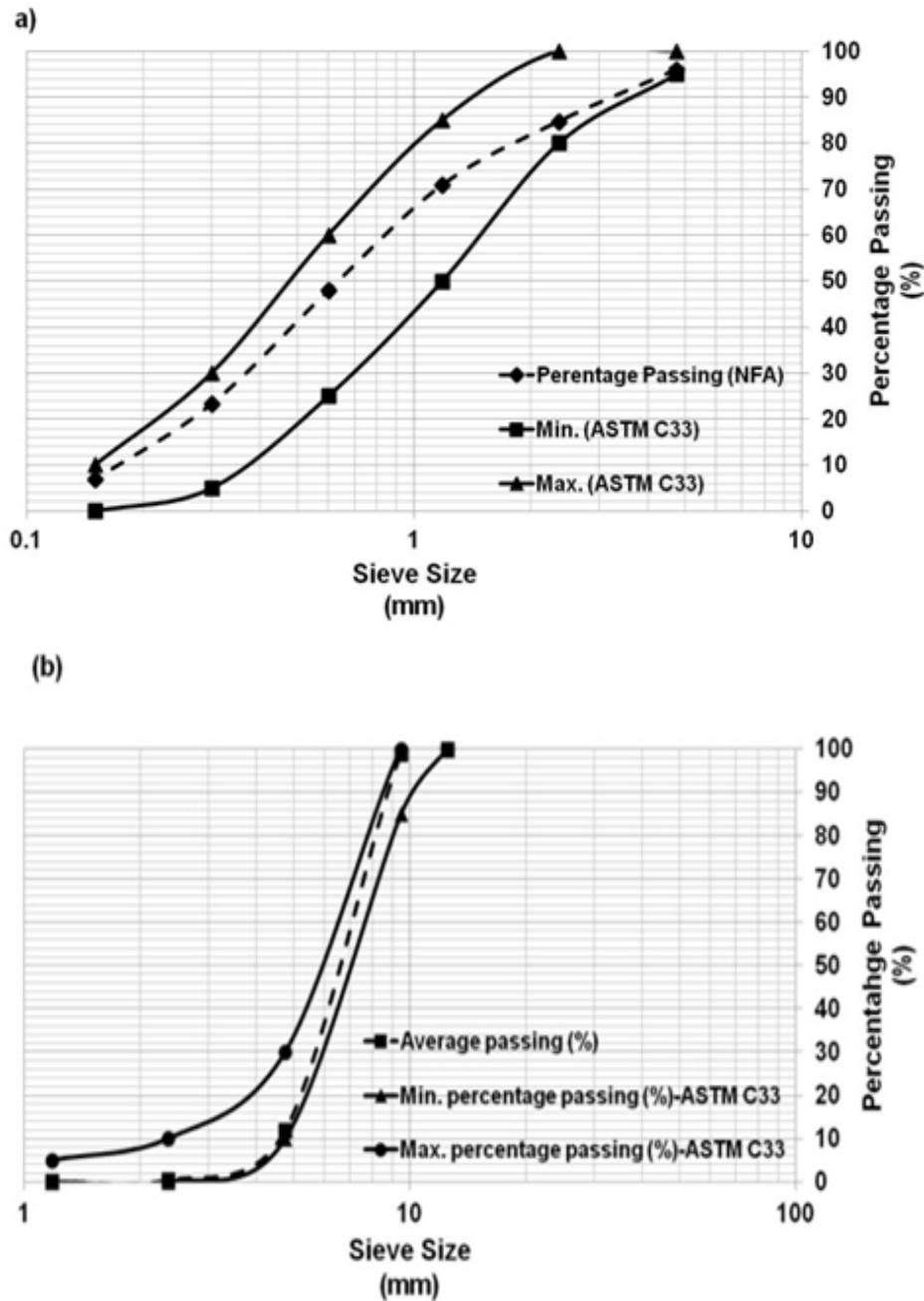


Figure 3.9 Sieve analysis of aggregate (a) Fine aggregate, and (b) Coarse aggregate

3.3.3.1.1 Mix Proportion

Mixtures differentiated based on the type and portion of agro-waste materials partially replace natural aggregate are represented in **Table 3-3** and **Table 3-4**. The 28 days target compressive strength for all mixtures was 30 MPa. All mixtures prepared according to ASTM C 192 (Standard

Practice for Making and Curing Concrete Test Specimens in the Laboratory). For each type of agro-waste materials (i.e., AWM1, AWM2, and AWM3), the coarse aggregate was replaced by agro-waste materials at rates of 0% 20% 30% and 40% by volume. For AWM4, it was added at rates 3 % 5% and 7% by volume total of the mixture. A water/cement ratio of 0.35 used for all mixtures.

3.3.3.1.2 Specimens Preparation

Concrete cylinders of Ø100mm×200mm were prepared to measure mechanical properties. All specimens were made and cured according to ASTM C192 “Standard Practice for Making and Curing Concrete Test Specimens in the Laboratory.” Compressive strength and indirect tensile strength were measured for a replicate of each mixture at different ages according to ASTM C39 “Standard Test Method for Compressive Strength of Cylindrical Concrete Specimens” and ASTM C496 “Standard Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens,” respectively.

Table 3-3 Mixture compositions for concrete incorporating agro-waste materials.

Materials	Mixtures composition (kg/m ³)			
	0% AWM	20% AWM	30% AWM	40% AWM
Cement	475	475	475	475
Coarse aggregate	1110	890	835	780
Fine aggregate	705	705	705	705
Water	165	165	165	165
AEA (ml)	5910	5910	5910	5910
W/C	0.35	0.35	0.35	0.35
Agro-Waste (AWM)				
AWM1	-----	6.10	7.50	9.15
AWM2	-----	50.00	63.00	75.00
AWM3	-----	15.50	19.00	23.00

Table 3-4 Mixture composition for concrete incorporating agro-waste fibres (AMW4)

Materials	Mixtures composition (kg/m ³)			
	0% AWM	3% AWM4	5% AWM4	7% AWM4
Cement	475	475	475	475
Coarse aggregate	1110	1095	1060	1035
Fine aggregate	705	705	705	705
Agro-Waste	-----	0.50	1.82	2.54
Water	165	165	165	165
AEA (ml)	5910	5910	5910	5910
W/C	0.35	0.35	0.35	0.35

3.3.3.1.3 Trial batches

Agro-waste material water absorption (as shown in figure 3.5) varies in the range of 2.18% to 5.49%. Hence, different additional water quantities added to trial mixtures incorporating 40% agro-waste materials to adjust flowability as follows: 20 ml, 250 ml, and 50 ml for mixtures incorporating AWM1, AWM2, and AWM3, respectively. Slump test values for tested concrete mixtures ranged from low (less than 25 mm) (**Figure 3.10a**) to moderate (70-95 mm) (**Figure 3.10 b**). At 0% replacement level (regular concrete), the slump values obtained were 80mm. For mixtures incorporating 20%,30%, and 40% of AWM2, the slump was, and 25, 20 and 15mm, respectively. Therefore, according to this preliminary result for workability, mixtures with AWM2 was eliminated from this phase as it had presumed weak subsidence. This result can be attributed to its high water absorption. Moreover, these mixtures replaced by two new concrete mixtures. In these two mixtures, AWM4 (i.e., fibre such as agro-waste) combined with two selected agro-waste materials (AWM1 and AWM3). **Table 3-5** presents a summary of the two new mixtures.



Figure 3.10 Slump test for mixtures with different agro-waste materials

Table 3-5 Mixture composition for concrete incorporating two types of agro-waste materials

Mixtures composition (kg/m³)

Materials	5% AWM4 + 35% AWM1	5% AWM4 + 35% AWM3
Cement	475	475
Coarse Aggregate	780	780
Fine aggregate	705	705
Water	165	165
AEA ml/100	5910	5910
W/C	0.35	0.35
AWM1	20.10	-----
AWM3	-----	8.10
AWM4	1.82	1.82

3.4 Results and Discussion

3.4.1 Fresh proprieties of concrete

Figure 3.11 shows the slump for mixtures incorporating various contents of agro-wastes. As mentioned earlier, concrete mixtures produced with AWM2 were not homogeneous and exhibited

a low slump value (around 65% reduction in slump value in comparison to the control mixture). As expected, slump values for these mixtures were found to decrease as the content of agro-waste AWM2 increased until concrete mixtures become unworkable (Figure 3.11). This result can be attributed to the difference between the granulometry of AWM2 and natural aggregate. Also, it is very high-water absorption regarding the nature of agro-waste, as observed in the chemical stability test, and changes in its shape had led to a very low slump in the trial mixtures and lead to remove AWM2 from our further examinations. This type of waste needs an in-depth study to be able to integrate it into concrete mixtures (i.e., pretreatment) or use it in particular types of concrete (i.e., zero-slump concrete) without comprising the overall performance.

Visual observation during mixing and compacting of all other concrete mixtures incorporating AWM1, AWM3, and AWM4 were homogenous, with a small amount of water added for each mixture according to the absorption rate of these materials. The slump values for these concrete mixtures were between 70 to 110 mm (**Figure 3.11- Figure 3.12**). Initially, mixtures incorporating 20% Agro-wastes AWM1 or AWM3 showed slightly higher slump values compared to control mixtures. At 30% AWM1 or AWM3, mixtures exhibited similar slump values to that of the control concrete mixtures. At 40%, there were reductions in slump values for all mixtures. This slightly higher and/or similar slump values for mixtures incorporating agro-waste can be attributed to the reduction in the amount of coarse aggregate. This had led to a higher paste volume in agro concrete mixtures regarding the amount of absorbed water and consequently increased their workability. The 30% agro-waste materials seem to be a threshold, above which the effect of water absorption and the angularity of particles will dominate the behaviour leading to a lower slump.

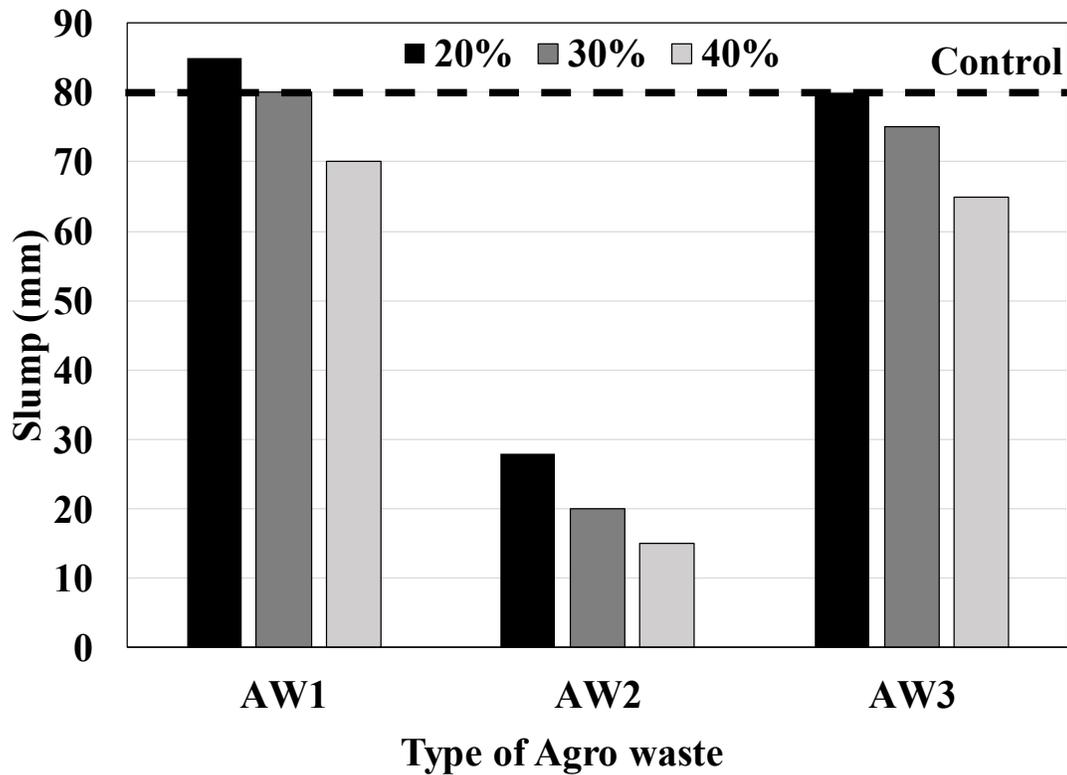


Figure 3.11 Development for concrete mixtures incorporating agro-waste materials.

For AWM4, lower agro-wastes contents used due to its fibrous shape. In general, the slump value increased as the AMW4 content increased. Mixtures incorporating 7% as a replacement of the total coarse aggregate exhibited the highest slump value (**Figure 3.12**). This also can be attributed to the increase in the paste volume by absorbed water agro-waste, which acts as a surrounding media for agro-waste to flow.

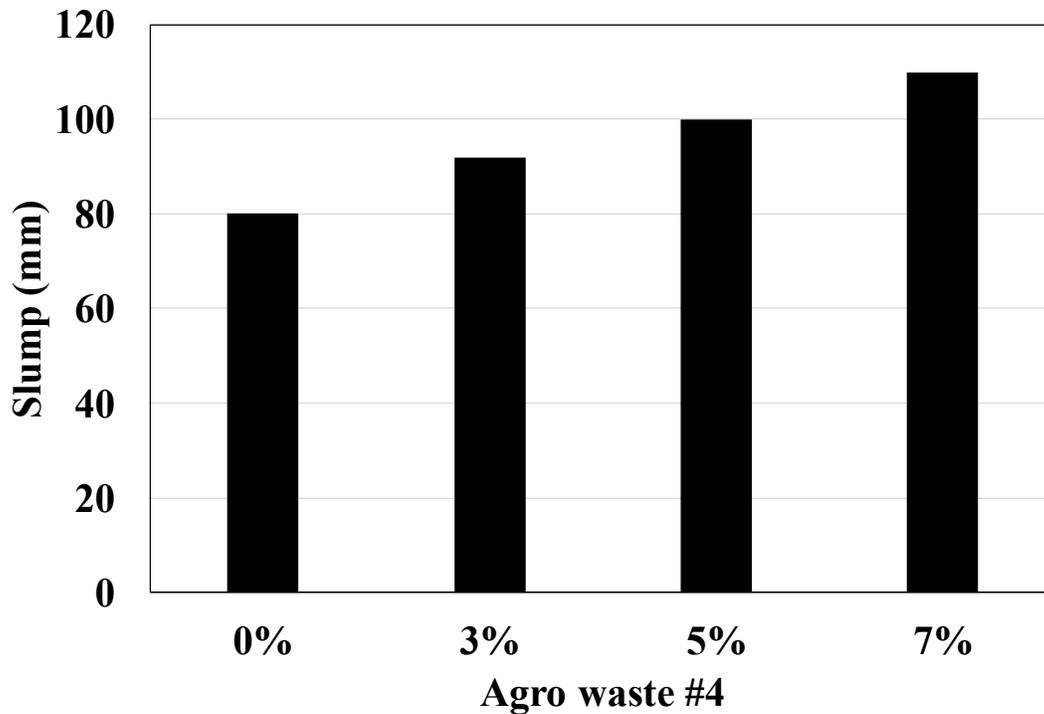


Figure 3.12 Slump development with different AWM4 percentages.

For mixtures incorporating 5% AWM4 combined with 35% of AWM1 or AWM3, the slump values varied (**Figure 3.13**). Combining AWM1 with AWM4 showed a slight improvement in the slump value compared to mixtures with 40% AWM1 only. This can be attributed to the low absorption of AWM4 compared to AWM4, which frees more water to assist in workability. Conversely, adding AWM4 to AWM3 did not induce any improvement. This can be related to two compensating factors: the increase in the fibrous content and an increase in free water. Agro-waste AWM3 has a portion of coarse particles; hence, adding AWM4 increases the fibrous content significantly, leading to a lower slump. On the other hand, AWM4 will free more water, as explained early, leading to a higher slump.

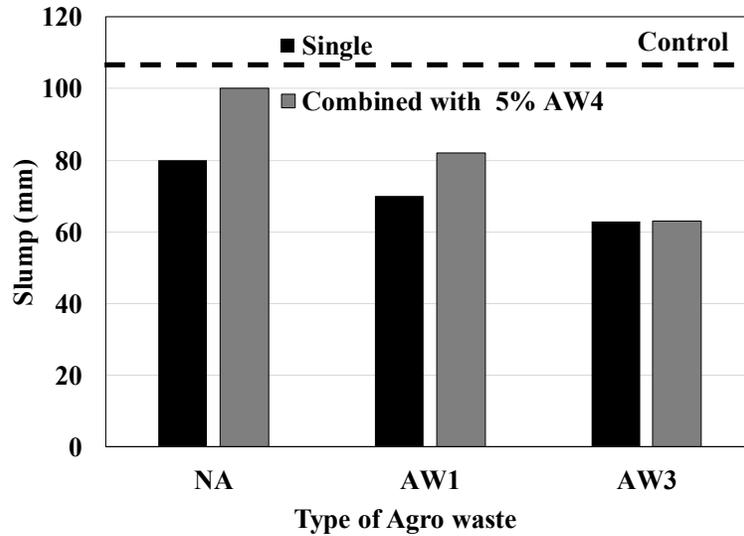


Figure 3.13 Slump for mixtures incorporating natural aggregate and agro-waste materials separately and combined.

3.4.2 Hardened Properties of Concrete

3.4.2.1 Compressive strength

Figure 3.14 and **Figure 3.15** show all tested mixtures compressive strength results with different contents of agro-wastes. For all mixtures, the compressive strength increased with time. For instance, the strength for AWM1 at the age of 28 days was about 56% higher than that at the age of 7 days. This increase in strength can be attributed to the progress in hydration and the development of more hydration products that form the concrete microstructure. Generally, the addition of agro-wastes was found to reduce the achieved compressive strength concerning that of control mixtures without agro-waste. However, reductions in compressive strength values decreased as the content of the agro-waste material increased up to a dosage of 30% as a replacement for natural aggregate. Above 30%, increasing the agro-waste content (i.e., 40%) results in lower strength. This can be attributed to different factors that compensate each other. These factors include water content, water absorption, paste volume, and agro-waste shape/size. Increasing the water to achieve the workability will increase the water-to-cement ratio of the mixtures leading to a lower strength. The high-water absorption for agro-waste materials will grantee high relative humidity at later ages, which will assist the progress in hydration reactions and strength gain. Increasing the paste volume will provide enough material to cover all the agro-

waste and developing an excellent bonding strength. However, increasing the paste volume will result in a lower solid aggregate content leading to a lower strength. Finally, shape and size for agro-waste materials with more fibre content (i.e., AWM3) will possess higher strength due to bridging cracks. Consequently, the achieved strength will increase as a result of restraining diagonal cracks formed under compression. Moreover, large agro-waste materials particles could provide a bridging effect (i.e., resembling the role of short fibre), leading to a better strength [174]. It is completely clear from the results that adding 30% agro-waste materials represents an optimum value at which a balance between all these compensating factors achieved.

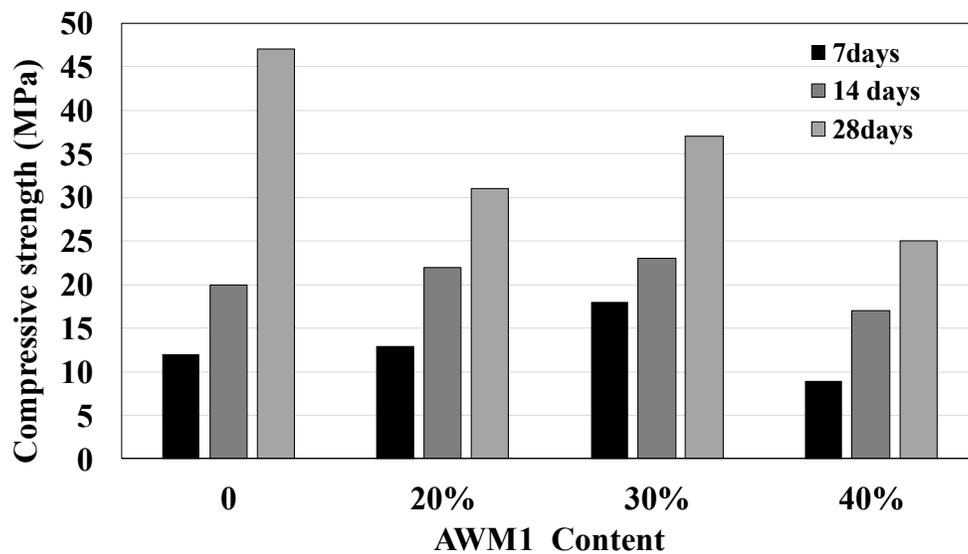


Figure 3.14 Compressive strength for concrete mixtures incorporating various contents of AWM1

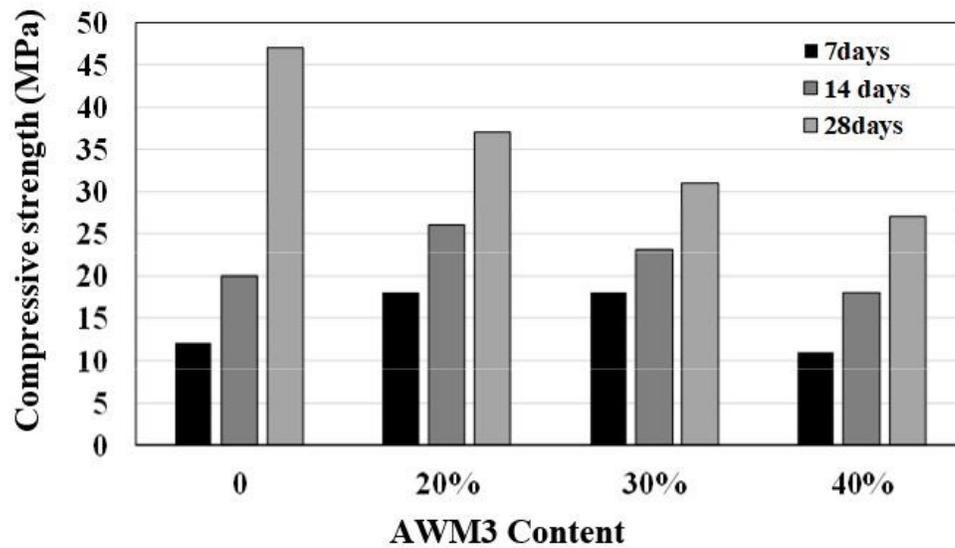


Figure 3.15 Compressive strength for concrete mixtures incorporating various contents of AWM3.

As illustrated in **Figure 3.16**, the rate of compressive strength loss decreases when increasing the replacement level of AMW4. The development of strength in natural fibre reinforced cement mostly depends on fibre's ability to bond to the matrix and/or each other. The bonding can be affected by dimensions, surface condition, absorption, and some fibre present in a given volume material[175]. Concrete mixtures incorporating 5% AWM4 exhibited comparable compressive strength to that of the control at 28 days. Above 5% AWM4, the strength decreased, which can be attributed to the increase in the fibres content leading to higher interlocking and voids.

Figure 3.17 shows the compressive strength for concrete mixtures incorporating AWM4 combined with AWM1 or AWM3. All mixtures had the same replacement rate of 40% by volume of natural aggregate. Results showed that combining two types of agro-waste materials improved compressive strengths compared to mixtures incorporating AWM1 or AWM3 alone. This can be ascribed to the fibre, which in turn, restrained micro-cracks.

Therefore, short and long fibres from agro-waste materials can be used in lightweight concrete applications. Furthermore, higher percentages of agro-waste materials can use in non-load bearing structures as a replacement for natural aggregate[176].

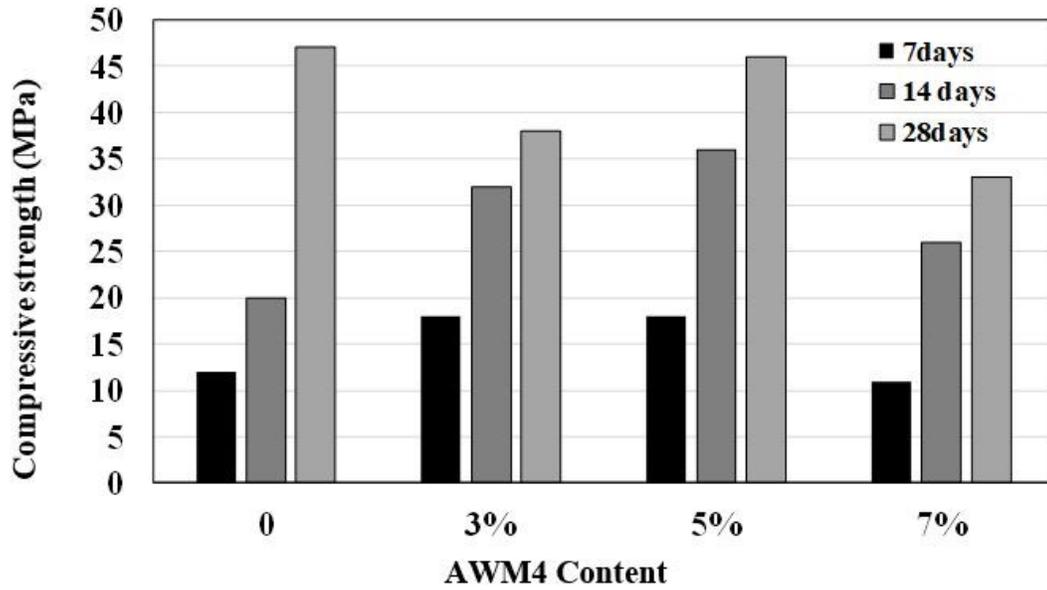


Figure 3.16 Compressive strength for concrete mixtures incorporating various contents of AWM4.

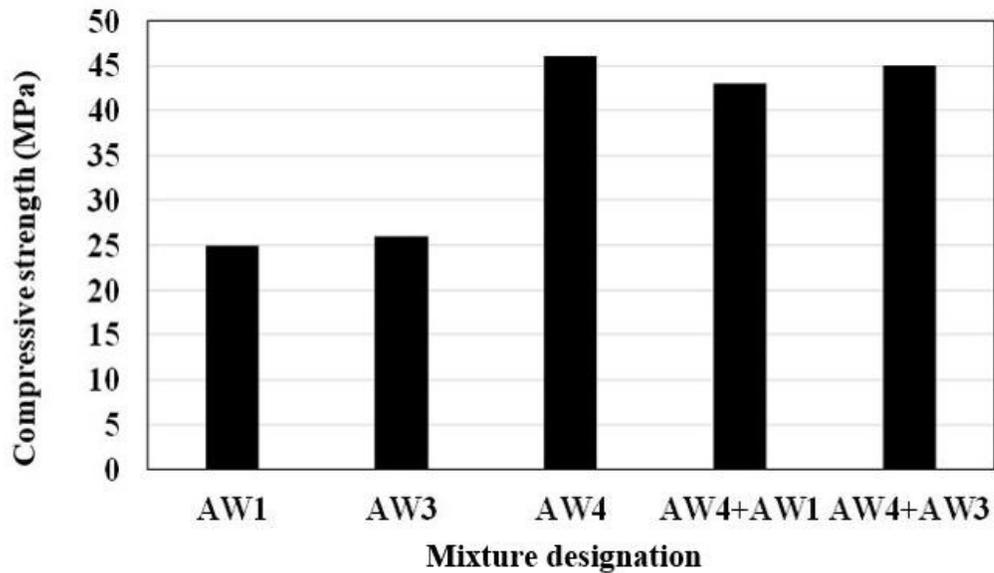


Figure 3.17 Compressive strength for concrete mixtures incorporating 40% agro-waste materials.

3.4.2.1 Splitting tensile strength test

Figure 3.18 shows the splitting tensile strength results for all concrete mixtures at the age of 28 days. Similar to compressive strength, adding agro-waste materials (i.e., AWM1 or AWM3) led to lower tensile strengths compared to that of the control mixtures. Moreover, increasing the agro-wastes contents to 30% resulted in a lower reduction in strength. At higher dosages above 30% agro-waste materials, tensile strength had decreased. It was interesting that AWM3 showed higher performance compared to AWM1. These results can be ascribed to the fact that AWM3 had a high portion of fibres. These fibres will probably assist in increased tensile strength. On the other hand, AWM4 had increased tensile strength. The higher the content, the higher the increase in tensile strength, as shown in **Figure 3.19**. This **Figure 3.19** can be attributed to the fibrous propriety, size, and surface morphology of agro-waste materials AWM4, which affect the mechanical properties of concrete positively and plays an essential role in reinforcing concrete with high efficiency[176].

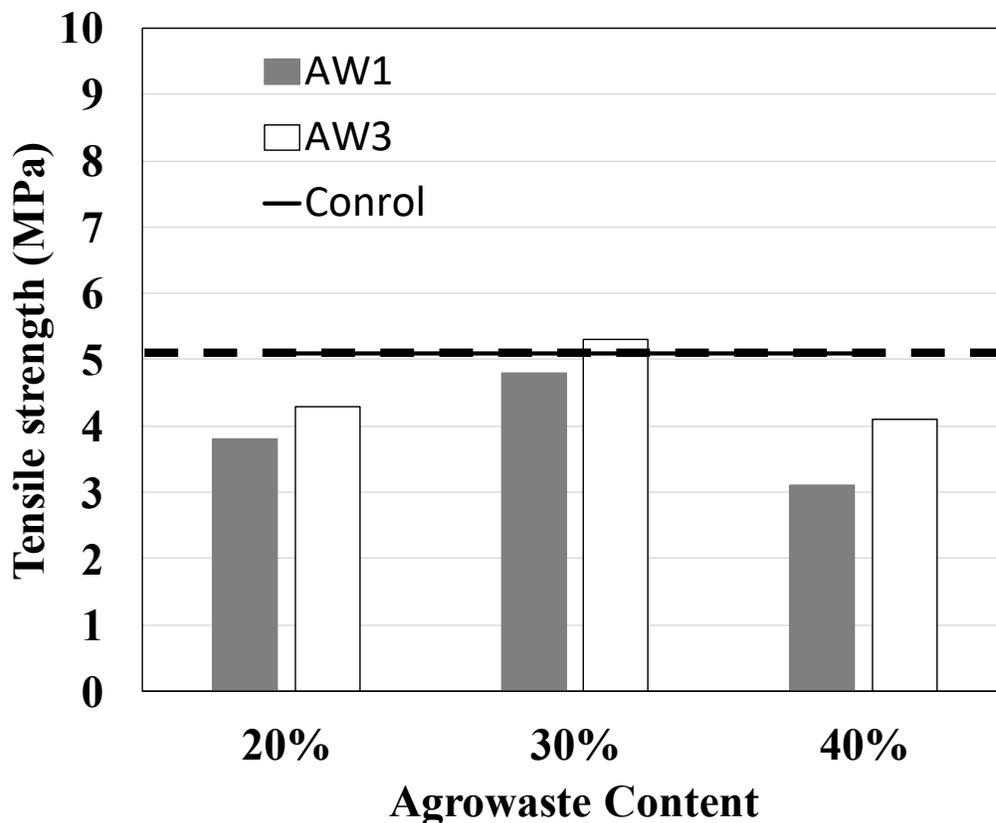


Figure 3.18 Tensile strength for agro concrete with various contents.

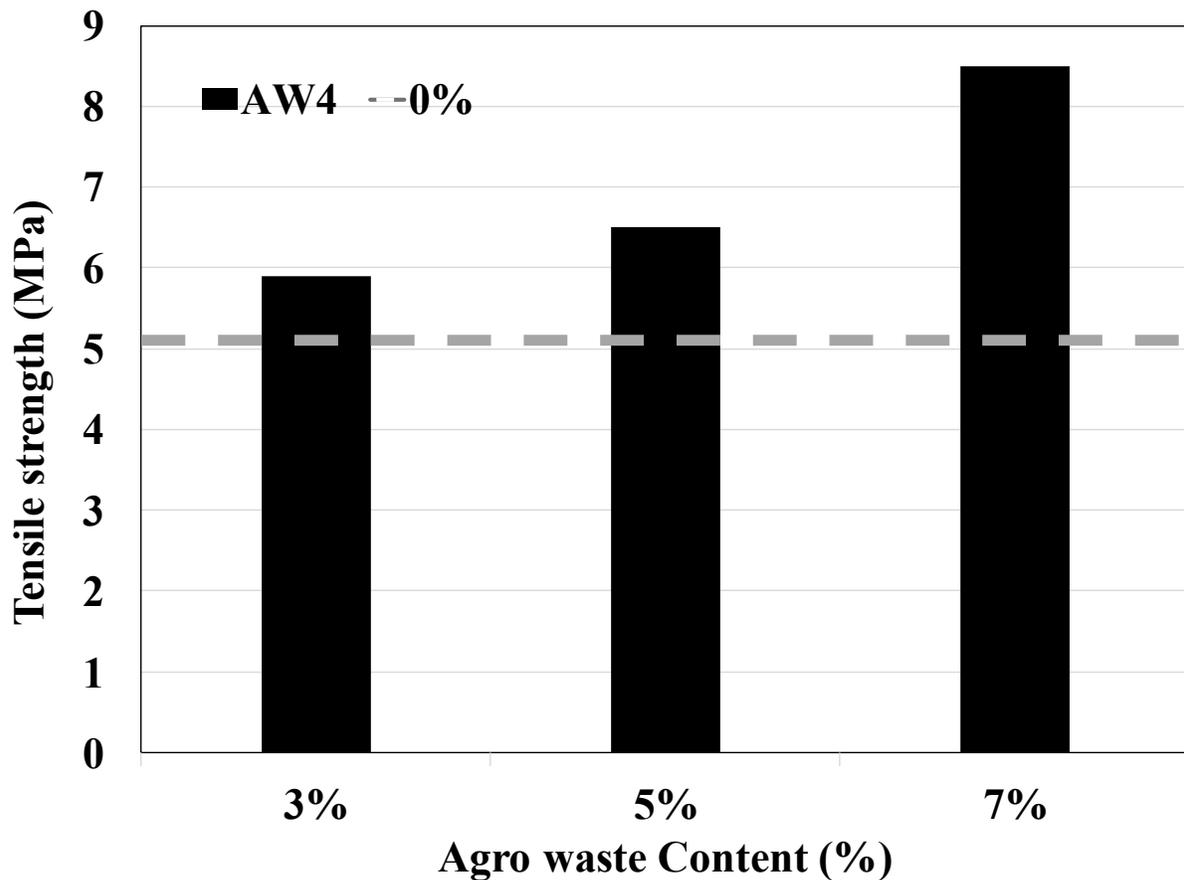


Figure 3.19 Tensile strength for agro concrete with various contents of AWM4

Figure 3.20 shows typical images of the different types of concrete made with agro-waste materials (AWM1, AWM3, AWM4, AWM1+AWM4, and AWM3+AWM4). The examined microstructure for AWM1 mixtures shown in **Figure 3.20 (a,b, and c)**. Results of the microstructure showed a weak bond between agro-waste materials AWM1 and mortar. Also, several surfaces and internal voids presented in the tested sample (**Figure 3.21**). This monitoring is in agreement with the reported results and explains the reductions in the compressive and tensile strength.

As shown in **Figure 3.20 e–I**, agro-waste particles and cement hydration products thoroughly integrated into the cement-bonded particle boards, which indicates good bonding at the interface of agro-waste-matrix. Moreover, the rupture of some agro-waste particles observed in the cross-section of fractured samples. This output implied that agro-wastes act as “fibre” under stress. The big particles act as short fibres (AWM1), while large particles act as fibrous materials. This result

explains the higher strengths exhibited by AWM3, which contained cell-fibres with a 15 mm diameter (**Figure 3.20 d,e, and f**).

Figure 3.22 shows the examined microstructure for mixtures incorporating two types of agrowastes (i.e., AWM4+ AWM1/AMW3). In the fractured particleboards with little wood fibre and long fibre, rough surfaces in the cross-section could be observed with mortar on the surface of the cellular fibres, indicating sufficient adhesion between the fibres and the mortar. Moreover, some traces of cell-fibres and remaining cell-fibres were observed after the tensile strength test, suggesting that fibre de-bonding, fibre siding, and crack bridging, which contributes to strength enhancement. Compared to the control mixtures, the arrangement of legitimate C–S–H gel observed in the zone (z_2) for mixtures AWM1+AWM4 (**Figure 3.22**). Therefore, the combination between two types of fibres in concrete will have a dual effect: AWM3 improves the bond between the aggregate and the cement matrix, and AWM4 maintains the bonds between the cement matrix and the fibre.

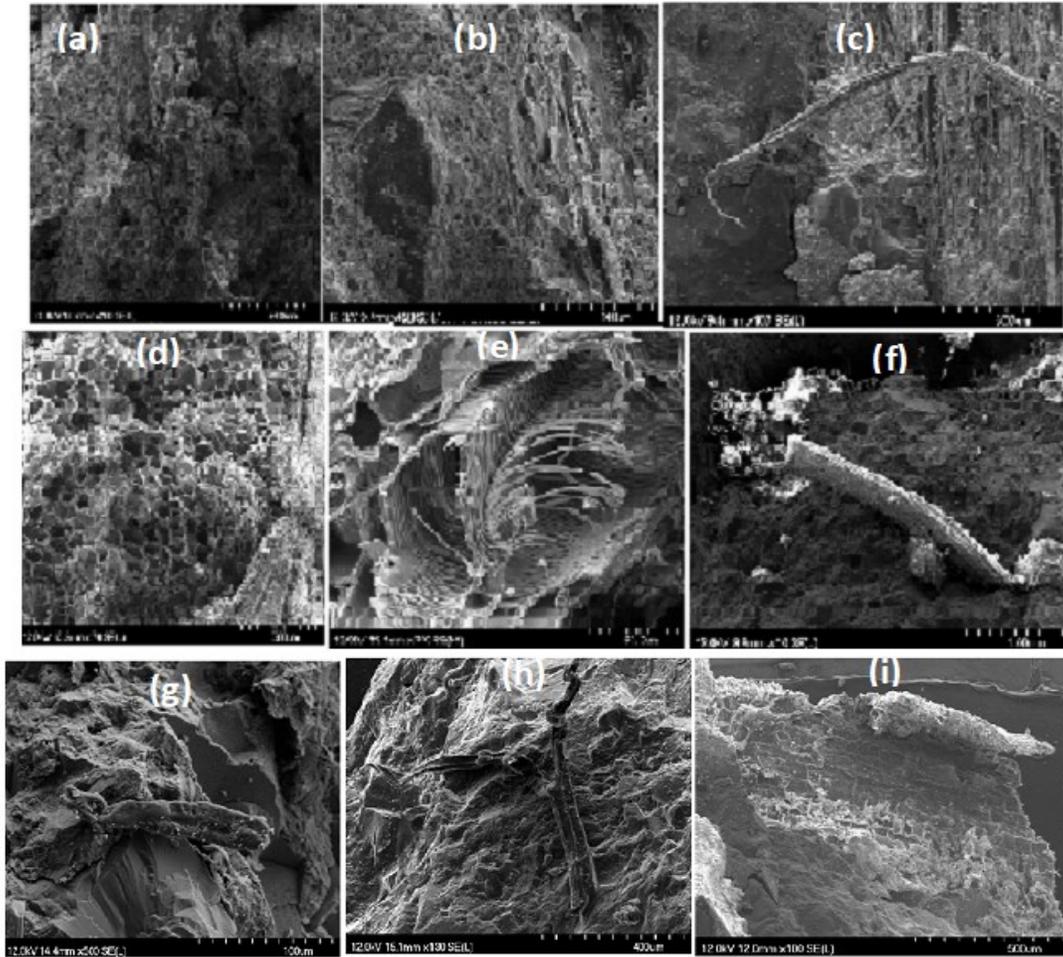


Figure 3.20 SEM images of fresh fracture of different sample concrete used the ago waste materials(a) AWM1-20%, (b) AWM1-30%, (c) AWM1-40%, (d): AWM3-20%, (e) AWM3-30%, (f): AWM3-40%, (g) AWM4-3%, (h) AWM4-5%, (i) AWM4-7%.

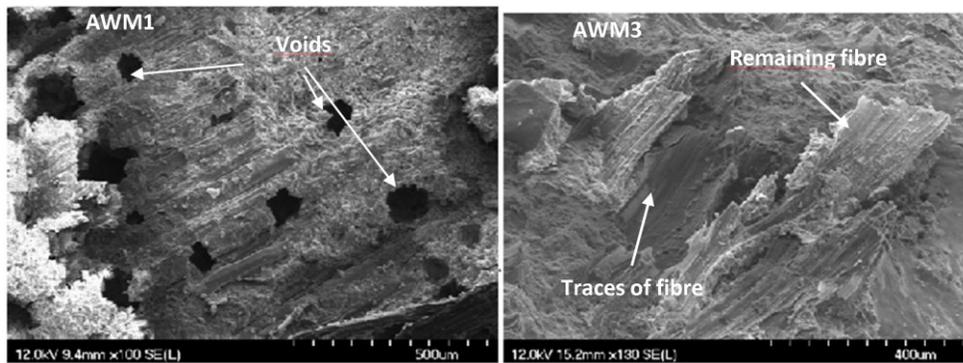


Figure 3.21 Microscope image of concrete made of agro-waste materials (AWM1 and AWM3).

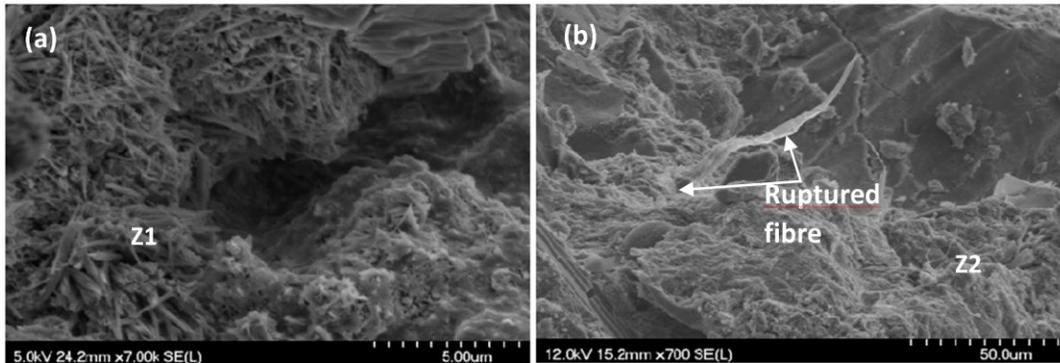


Figure 3.22 Microscope image of concrete made of agro-waste materials: a) 35%AWM1+5%AWM4, (b) 35%AWM3+5%AWM4. Z1 fewer ettringite needles, Z2: C-S-H gel

3.5 CONCLUSION

This chapter focused on examining the potential of using different agro-waste materials to produce green construction materials. The initial phase focused on the evaluation of the properties of agro-waste materials, followed by different experimental work phases. The most relevant results can be summarized as follow:

- Properties of agro-waste materials is a dominant factor in selecting the suitable applications and method of implementation in construction materials.
- Combining more than one agro-waste material has a significant benefit in sharing their different features and overcome drawbacks of a specific type.
- Agro-waste concrete can be produced with different strength grades based on the amount of agro-waste materials included.
- Agro-waste materials showed high potential as filler materials in particular concrete types.
- Particular types of concrete can be designed to maximize the benefits of adding agro-waste materials.

CHAPTER 4. PROPERTIES OF BIO BASED CONTROLLED LOW STRENGTH MATERIALS

This study investigates the feasibility of enhancing construction material's sustainability using bio-based by-products (i.e., agro-waste) as a partial replacement for natural sand. Controlled low strength material (CLSM) was selected as a practical application due to its low strength requirements. Fine agro-waste was added at different strength levels CLSM mixtures at rates 0%, 5%, 10%, and 20% as a partial replacement of natural sand. Fresh and hardened properties, including flowability, bleeding, density, compressive, and tensile strengths, were evaluated. Agro-waste CLSM mixtures were exposed to a sulphate environment to examine their durability. The experimental results demonstrate that adding agro-waste had reduced the compressive strength; however, this reduction was within the acceptable limit.

Moreover, results indicated that mixtures with high cement content could incorporate more than 20% agro-waste while maintaining adequate performance. The fine agro-waste has some filling effect and can reduce the pores leading to lower absorption rates. Dry/wet cycles will adversely affect the volume stability of CLSM, forming wide cracks that, in turn, accelerate degradation. Producing CLSM for farm applications is a feasible method for reusing such waste.

*A version of this chapter is under review at Construction and Building Materials Journal (submission ID #CONBUILDMAT-D-19-08664)

4.1 Introduction

Concrete is the most used construction material [147]. A wide range of constructional applications, including bridges, roads, tunnels, and various buildings are mainly concrete. Concrete production is expected to grow to approximately 18 billion tons by 2050 [177]. Accordingly, a considerable amount of natural aggregates, about 60 to 70% of that amount of produced concrete, is going to be consumed, leading to scarcity of resources. This has motivated researchers to find alternative sources for natural aggregates. Various solid wastes generated by different sectors such as industrial, mining, construction, and agricultural activities had been tested [62, 178-180]. Using agriculture wastes as aggregate has attracted many researchers targeting a low carbon, sustainable, and environmentally friendly concrete [2, 181-183]. The availability of agricultural wastes makes them a suitable and dependable alternative for natural aggregate in concrete. The amount and type of agricultural wastes vary between countries according to the type of crops and farming land [2]. Globally, farmers used to get rid of such waste by either burning it or storing it in the roofs of their houses, which increases fire risk along with harming the environment. Therefore, in many cases, reusing and recycling such wastes in cementitious materials considered a cost-effective, sustainable solution for both agriculture and construction sectors. Several agricultural wastes such as hemp, coconut shells, and others were utilized successfully in producing structural and non-structural cementitious materials [182, 184, 185]. However, reduction in strength was a common observation which halts using of such waste as a replacement for aggregate in conventional concrete. With the advent of controlled low strength materials (CLSM), the potential for utilizing agriculture wastes as aggregate has increased [8].

According to the American Concrete Institute (ACI), CLSM is a self-compacting cementitious material that is primarily used to replace conventional backfill soil [186]. It can be considered as a structural backfill [131, 141]. CLSM was known by different names, such as flowable fill or mortar, soil-cement slurry, and plastic soil-cement. The unconfined compressive strengths for CLSM is around 8.3 MPa or less [141]. If future excavation anticipated, the maximum later compressive strength should not exceed 2.1 MPa [186]. This relatively low strength is the main advantage of CLSM, allowing the incorporation of high amounts of solid wastes. Many materials have used in CLSM as substitute fine aggregates such as boiler slag, cement kiln dust, chip ballast, treated oil sands waste, and other similar industrial and mining wastes [131, 187, 188]

Hence, this study aims to provide engineering properties of Bio-CLSM incorporating agriculture wastes. The successful use of such agriculture wastes, as full or partial replacement of natural aggregate, will lead to mutual benefits for both construction and agricultural sectors, including saving energy and natural resources, reducing the cost of construction materials, solving disposal problems associated with agriculture wastes. Moreover, the findings will pave the way for other potential uses of agricultural wastes in the construction industry, targeting a more environmentally sustainable concrete industry.

4.2 Experimental work

4.2.1 Material

General used (GU) hydraulic cement according to the CSA-3001 with Blaine fineness of $360 \text{ m}^2/\text{kg}$ and a specific gravity of 3.15, and Class F fly ash (FA) according to ASTM C618 were used as binding materials for all tested CLSM mixtures. **Table4-1** shows the chemical and physical properties for cement and fly-ash. Natural river sand with a specific gravity of 2.65 and particle size distribution complying with that of fine aggregate for concrete, according to ASTM C33, was used. The used waste (i.e., which will be named so after “agro-waste”) was a fine residual from black spruce with a specific gravity 0.41. It had a micro-size particle along with some micro size fibrous shaped particles. It should be mentioned that black spruce grows in a broad transcontinental band from Alaska (United States) to Newfoundland (Canada) [189]. It accounts to accounts for approximately 12% of Canada’s total softwood inventory. The disposal of such residues is a problem for the forest industry. However, it had been implemented in different applications, but a significant amount of bark is still unused[190]. In this study, three control mixtures with different cement contents (i.e., 30 kg/m^3 , 60 kg/m^3 , and 90 kg/m^3) were prepared based on proportion guidelines reported by the ACI committee 229[186]. The same mixtures were modified to incorporate agro-waste as partial replacement of sand by volume at rates of 0%, 5%, 10%, and 20%. Mixture proportions are shown in **Table4-2**.

Table 4-1 Chemical and physical properties of cement and fly ash.

		GU	FA
SiO ₂	(%)	19.80	43.39
Al ₂ O ₃	(%)	4.90	22.08
CaO	(%)	62.30	15.63
Fe ₂ O ₃	(%)	2.30	7.74
SO ₃	(%)	3.70	1.72
K ₂ O	(%)	0.83	--
Na ₂ O	(%)	0.34	1.01
MgO	(%)	2.80	-----
P ₂ O ₅	(%)	0.11	-----
Na ₂ O _{eq}	(%)	0.87	-----
Loss on ignition	(%)	1.90	0.58
Specific gravity	--	3.15	2.50
Surface are	(m ² /kg)	360	280

4.2.2 Mixing procedure

The mixing procedure was conducted following relevant work in the literature [131, 191]. Initially, dry mixture components (i.e., cement, fly ash, and agro-waste) were mixed without water addition for 1 min to ensure a homogeneous distribution. Mixing water was then divided into two halves. The first half of the mixing water was added gradually to the mixture while continuing mixing for one more minute. The second half was then added and mixed for another 1 minute. The mixture was allowed to rest for 1 min after adding the whole amount of mixing water. Then, the mixture was mixed for an additional 2 min before sampling. For all tested mixtures, the flowability was measured continuously with various mixing water additions targeting the desired normal flowability in the range of 150 mm to 200 mm as recommended by [186].

4.2.3 Testing

Effects of agro-waste addition on CLSM fresh properties, including flowability, unit weight and bleeding, were evaluated following ASTM standards D6103-04 (Flow Consistency of Controlled Low Strength Material), ASTM D6023-07 (Density, Yield, Cement Content, and Air Content (Gravimetric) of CLSM) and ASTM test method C232 (Standard Test Method for Bleeding of Concrete), respectively. Cubic specimens 50 × 50 × 50 mm was used to evaluate the compressive strength for CLSM mixtures incorporating different percentages of agro-waste according to the ASTM test method D4832-10 (Standard Test Method for Preparation and Testing of CLSM Test Cylinders). The compressive test was conducted using a strain-controlled unconfined compressive strength machine at ages 7, 14, and 28 days. Specimens were kept inside the mold uncovered inside a curing room (temperature $22 \pm 2^\circ\text{C}$ and relative humidity $95\% \pm 3\%$) until the testing age due to the insufficient early-age strength of CLSM mixtures.

Table 4-2 Mixture compositions for CLSM mixtures with agro-waste materials

Mix Code	Materials (kg/m ³)				
	Cement	Fly ash	Sand	Agro-waste	Water
G30	30	148	1725	----	297
G30W5	30	148	1640	10	302
G30W10	30	148	1555	20	303
G30W20	30	148	1380	40	306
G60	60	148	1690	----	297
G60W5	60	148	1605	10	266
G60W10	60	148	1520	20	270
G60W20	60	148	1350	40	279
G90	90	148	1655	----	297
G90W5	90	148	1570	10	278

Mix Code	Materials (kg/m ³)				
	Cement	Fly ash	Sand	Agro-waste	Water
G90W10	90	148	1490	20	282
G90W20	90	148	1325	40	290

Moreover, splitting tensile strength was conducted for CLSM mixtures at the age of 28 days according to ASTM standards C496/C496M (Standard Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens). Prismatic specimens 25 mm × 25 mm × 280 mm were used to evaluate shrinkage for CLSM incorporating agro-waste. Specimens were kept for seven days inside the mold in plastic bags to avoid breakage and to reduce water evaporation from the surface. After demolding the specimens, initial readings were taken and then stored at lab condition (temperature 22 ± 2°C and relative humidity 50% ± 3%) until the testing age. The readings were taken daily until no change was recorded.

Ultrasonic pulse velocity was also conducted on CLSM to examine the density variation inside a CLSM and detect any internal defects. The test was conducted according to ASTM C597 (Standard Test Method for Pulse Velocity Through Concrete). Also, porosity content and connectivity were evaluated based on the absorption rate for CLSM specimens, according to ASTM C642 (Standard Test Method for Density, Absorption, and Voids in Hardened Concrete). Sulphate attack resistance was evaluated following the same procedure conducted by [141]. At age 28 days, specimens were exposed to dry/wetting cycles. Each cycle consisted of specimens drying in an oven at 100 ± 5 °C for 24 h and then soaked in very high concentration sodium sulphate solution (i.e., 350 g of the anhydrous sodium sulphate per 1 L of water) at 22 °C for 24 hours. Specimens' changes in residual strength were monitored with the number of cycles.

4.3 RESULTS AND DISCUSSION

4.3.1 Flowability

Figure 4.1 shows flowability results for all tested CLSM mixtures. For all mixtures, the water content was adjusted to achieve an adequate flowability within the range of 150-200 mm. The flow values were in the range from 151 to 189 mm, which falls within the normal flowability category according to [186]. At the same water content, increasing the cement content while maintaining the

same fly ash content reduced the flowability. For instance, increasing the cement content from 30 kg/m³ to 90 kg/m³ had reduced the flow from 182 mm to 151 mm. However, all mixtures were still within the acceptable range.

On the other hand, replacing sand with the agro-waste reduced the required amount of water slightly to achieve the same flowability range of control mixtures (**Figure 4.1**). This can be attributed to two compensating effects induced by the fine agro-waste: increasing water demand and freeing entrapped water. Adding such fine agro-waste increases the surface area of the particles in the mixtures leading to higher water demand. Simultaneously, the fine particles of agro-waste can fill voids between binder particles and release the entrapped water[131]. Consequently, more free water is available for lubrication and enhancing the flowability for the tested CLSM mixtures. Also, these fine agro-waste particles act as a lubricant between coarser particles (i.e. sand), leading to a lower particle's interferences and, consequently, the viscosity[192]. This explains the behaviour for mixtures incorporating 20% agro-wastes, which maintained the same flowability or slightly higher compared to those mixtures without agro-waste while possessing a lower w/s ratio.

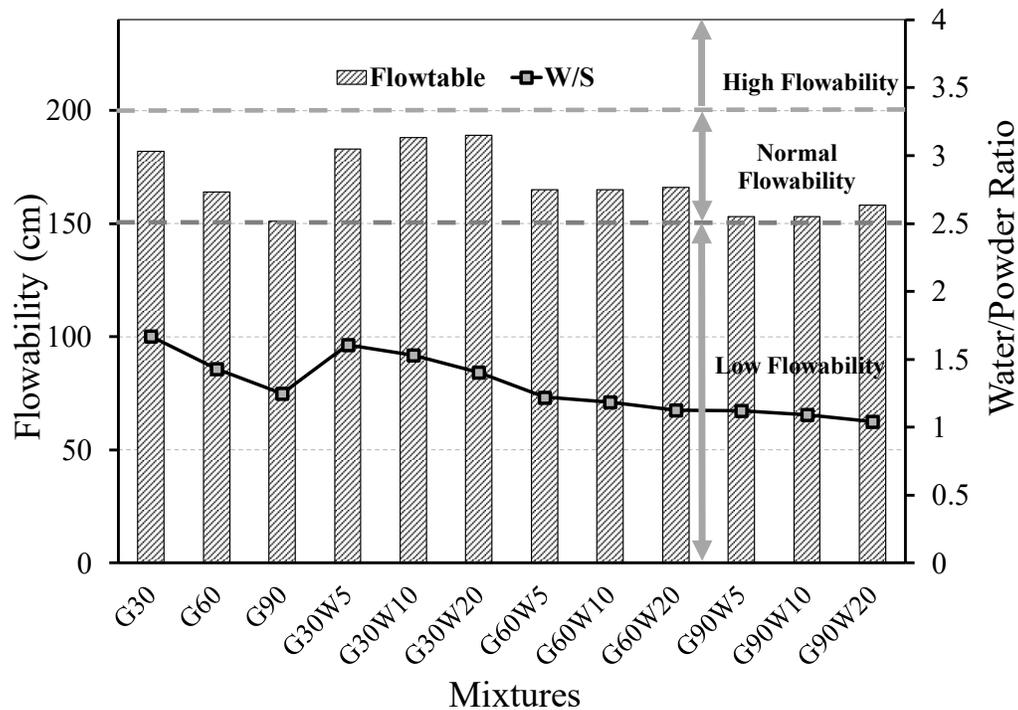


Figure 4.1: Flowability results for all tested CLSM mixtures.

4.3.2 Bleeding

Figure 4.2 shows the bleeding measured for all tested CLSM mixtures. All mixtures were stable and did not exceed the bleeding limit (maximum of 5% for stable CLSM [193]). As expected, increasing the cement content had reduced the bleeding. This is ascribed to the high consumption of water in absorption. Hence, less free water is available for bleeding[131]. For instance, control mixtures (G90) with 90 kg/m^3 exhibited around 50% less than that of mixtures (G30) that incorporated 30 kg/m^3 . Mixtures incorporating agro-waste materials exhibited lower bleeding than that for mixtures without agro-waste materials. For example, the reduction in bleeding was in the range of 26% to 63% for G60 as agro-waste content increased from 5% to 20%. This reduction can be attributed to the fact that the incorporation of fine agro-waste increases the amount of water needed to cover the fine particles, which keeps water from escaping to the surface as bleed water during the setting of the mixture[194]. One interesting point, increasing the waste content from 5% to 20% did not affect the bleeding for mixtures incorporating 30 kg/m^3 . This can be attributed to the high-water content of these mixtures. Therefore, even after the consumption and absorption of water on the surface of the agro-waste particles, there was still enough free water for bleeding.

However, all bleeding values for all CLSM with and without agro-waste were within the acceptable limit a, mentioned earlier[193].

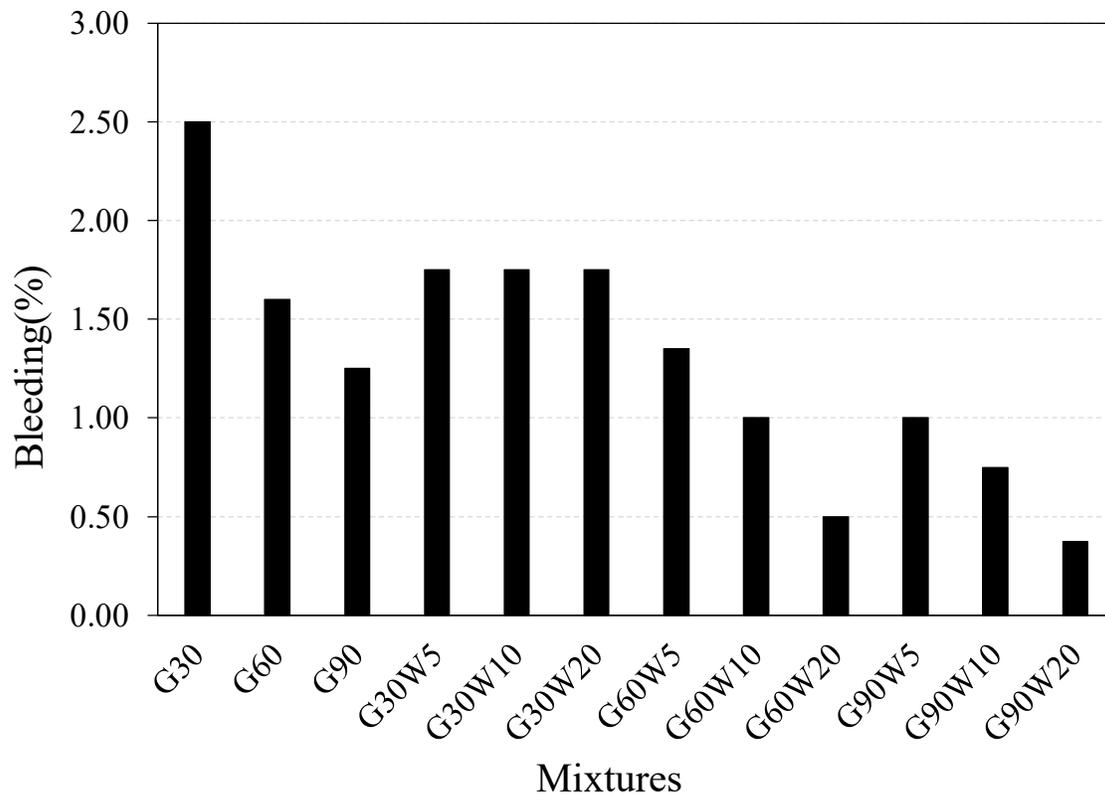


Figure 4.2 Bleeding results for all tested CLSM mixtures

4.3.3 Density

The density of the fresh and hardened CLSM samples was measured. Fresh densities for control mixtures ranged from 2085 kg/m³ to 2196 kg/m³, meeting the normal range for CLSM reported by ACI Committee 229[186]. For mixtures incorporating different percentages of agro-wastes, the fresh density decreased. The higher the percentage of agro-waste, the lower the density. For instance, mixtures with 60 kg/m³ cement, adding 5%, and 20% of agro-waste resulted in around 8% and 13% reductions in the fresh densities compared to that of the control mixture without agro-waste, respectively. The reduction in fresh density can be attributed to the low specific gravity of agro-waste compared to that of the natural sand. Despite these reductions in the fresh densities, all mixtures were still within the range for normal CLSM (i.e., 1842 kg/m³ to 2323 kg/m³) according to ACI Committee 229[186], except G30W20 mixture which was marginally (i.e. only 10 g) below

the lower limit. For all tested mixtures, dry density measured at age 28 days was lower than that of the fresh density, which is attributed to the water loss from specimens.

Moreover, dry density followed the same trend of fresh density. All dry densities were within the range for normal CLSM (i.e. 1762 kg/m³ to 1890 kg/m³), according to ACI Committee 229[186], except G30W20, which was below the lower limit. It should be mentioned that these results pave the way for further investigation at which higher percentages of agro-waste (i.e., > 20%) can be added to achieve lightweight CLSM with density and strength as low as 288 k/m³ and 68.95 kPa, respectively.

4.3.4 Compressive Strength

Figure 4.3 summarizes the compressive strength results for tested CLSM at ages 3, 7, and 28 days. Generally, the lower the cement content, the lower the achieved strength. This is attributed to the fact that reducing cement content lowers calcium hydroxide (CH) in the mixture. Consequently, the rate of pozzolanic reactions between silicate, from fly ash, and CH to form calcium silicate hydrated (CSH) (i.e. which acts a binding material) becomes slower [195]. As a result, the strength gain rates for G30 and G60 mixtures were expected to be lower than that of the G90 mixtures.

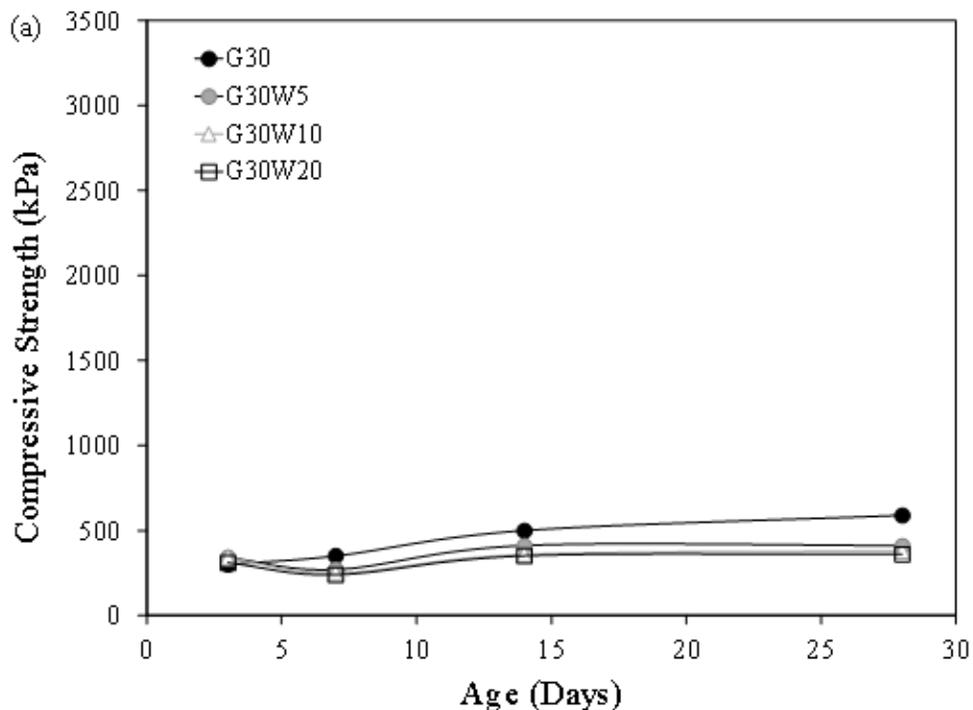
For mixtures incorporating agro-waste materials, it was clear that the strength had decreased as the amount of agro-waste increased. Moreover, the effect of agro-waste on strength was evident in rich mixtures (i.e. high cement content, 90 kg/m³) (**Figure 4.3c**). Conversely, for lean mixtures (i.e. low cement content 30 kg/m³), the variations in strength due to agro-waste addition were insignificant (**Figure 4.3a**). Hence, the higher the cement content, the higher the reduction in strength induced by agro-waste addition. This can be ascribed to two reasons: the high water/powder ratio and low CH in lean mixtures. However, increasing the cement content still showing an excellent solution to overcome the reduction in strength if required. For example, increasing the cement content from 30 kg/m³ to 90 kg/m³ for mixtures incorporating 20% agro-waste led to an increase in the achieved compressive strength of about 300% (i.e., from 360 kPa for G30W20 mixture to 910 kPa for G90W20 mixture).

The relatively low strength of CLSM fits with many of its applications. For instance, maintaining a low strength is very important to facilitate future excavation of such filling materials. According to the ACI committee 229[186], it is recommended that the compressive strength for CLSM to

lower than 0.7 MPa if future excavation is anticipated. The removability modulus (RE) can be used to assess the excavatability of a CLSM mixture based on its strength and dry density (**Eq. 1**).

$$RE = \frac{W^{1.5} \times 0.619 \times C^{0.5}}{10^6} \quad \text{Eq1}$$

Where W is the dry density of the mixture in (kg/m^3), C is the compressive strength at 28 days in (kPa). The CLSM mixture is considered easily removable if RE is less than 1 [186]. **Figure 4.4** shows the RE values for all tested mixtures. Adding agro-waste materials for lean mixtures (i.e. cement content 30 kg/m^3 and/or 60 kg/m^3) enhanced their removability. For instance, at 20% agro-waste materials, RE values were 0.65 and 0.83 for mixtures with 30 kg/m^3 and 60 kg/m^3 , respectively. However, for mixtures with 90 kg/m^3 , RE values are above 1, even at 20% agro-wastes. This indicates the capability of G90 mixtures to accommodate higher contents of agro-waste while maintaining the excavatability requirements.



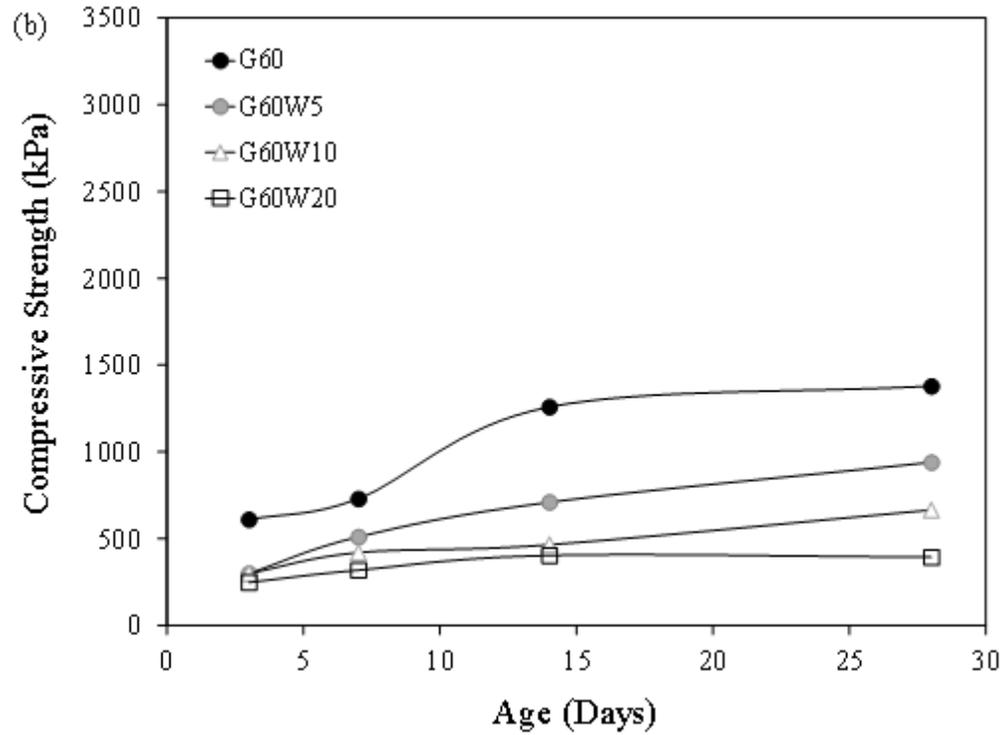


Figure 4.3 Compressive strength development for tested CLSM with cement contents a) 30 kg/m^3 , b) 60 kg/m^3 and c) 90 kg/m^3 .

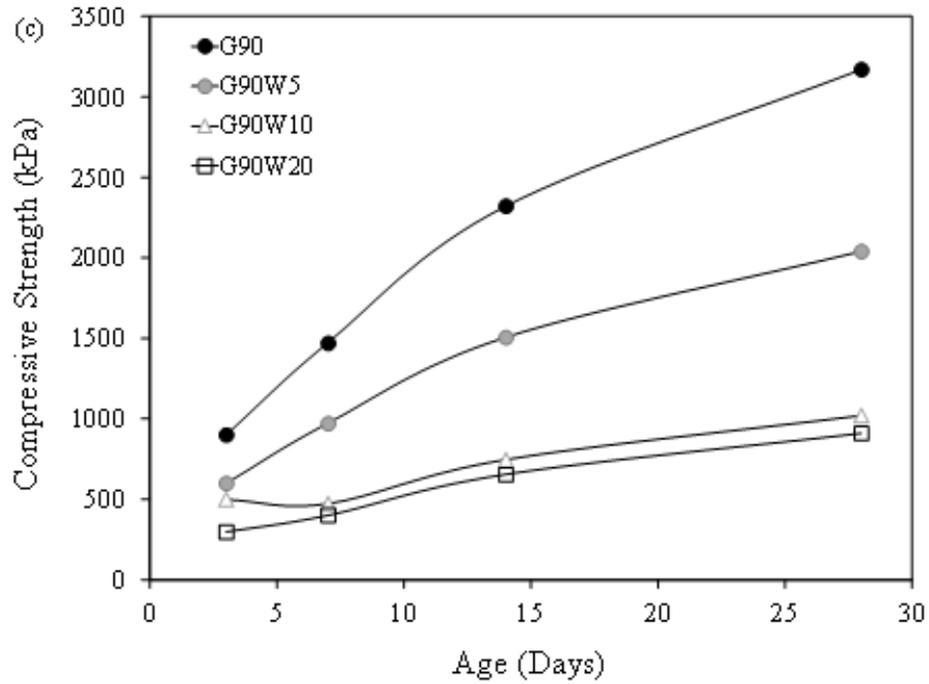


Figure 4.4 Compressive strength development for tested CLSM with cement contents a) 30 kg/m³, b) 60 kg/m³ and c) 90 kg/m³.

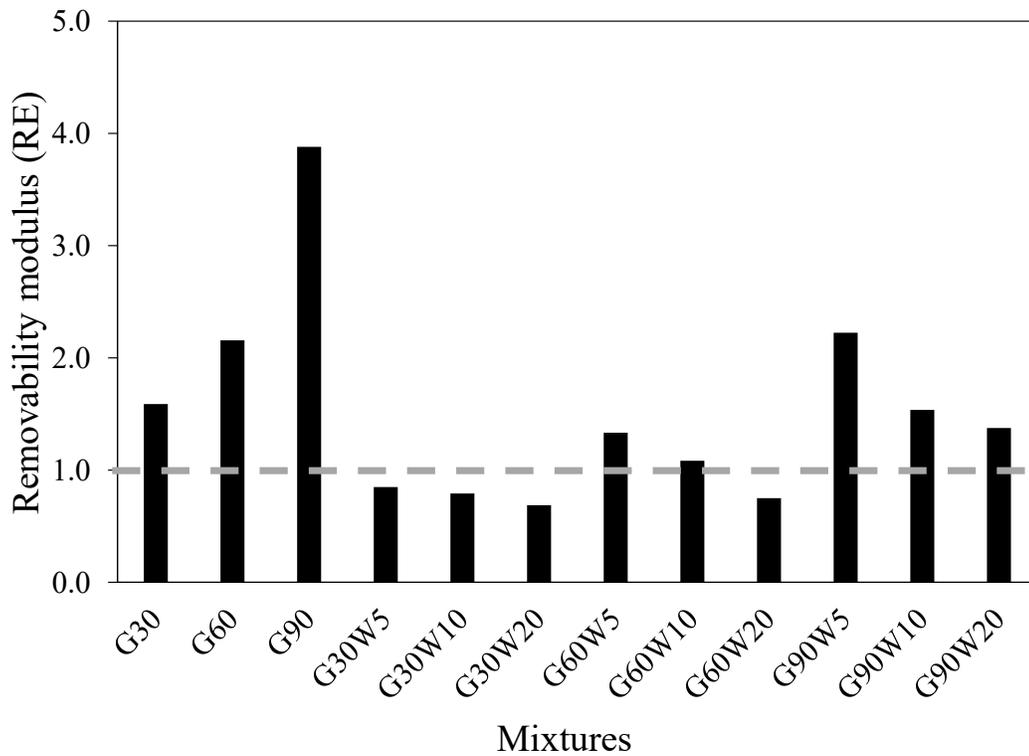


Figure 4.5 RE values for all tested CLSM mixtures.

4.3.5 Splitting Tensile Strength

The splitting tensile strengths for the concrete mixtures are presented in **Figure 4.5**. The control mixtures without agro-wastes achieved tensile strength in the range of 340 kPa to 1190 kPa at the age of 28 days. The measured splitting tensile strength of the concrete mixtures containing agro-waste generally followed the same pattern as compressive strength. Similar to the compressive strength trend, tensile strength decreased as the amount of agro-waste increased in the mixtures. Moreover, a good linear relationship between the tensile strength and the compressive strength of the tested CLSM samples (**Figure 4.6**). This agreed with previous studies on CLSM[131, 196].

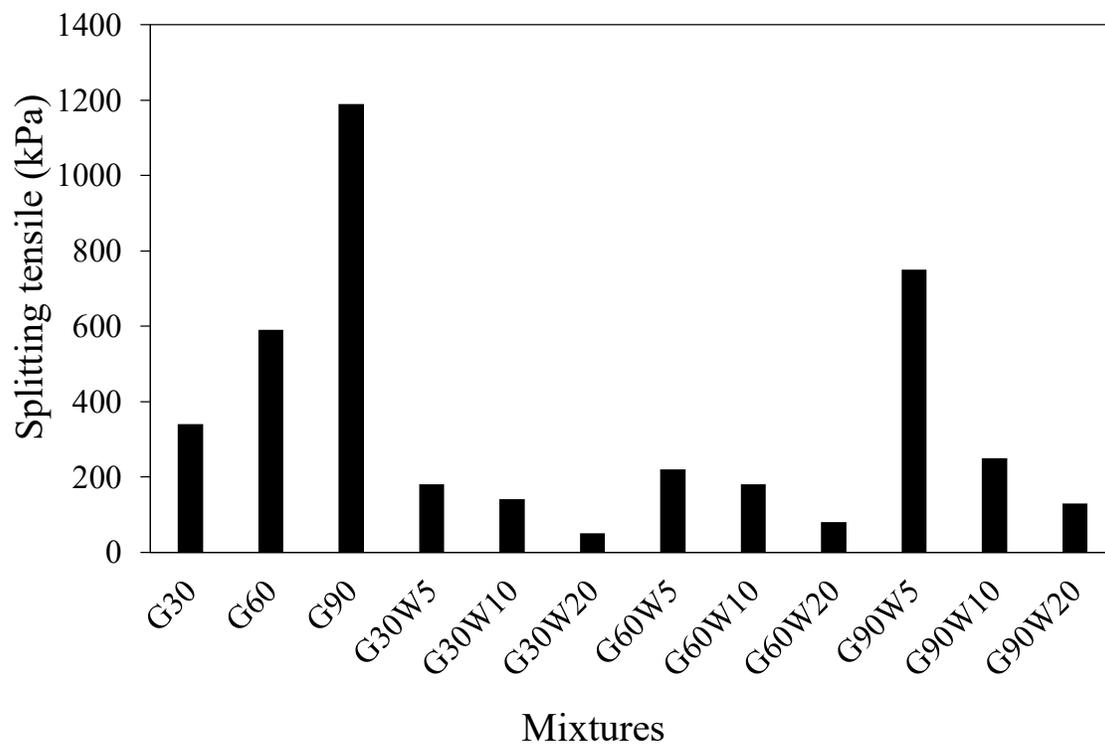


Figure 4.6 Splitting tensile strength for all CLSM mixtures.

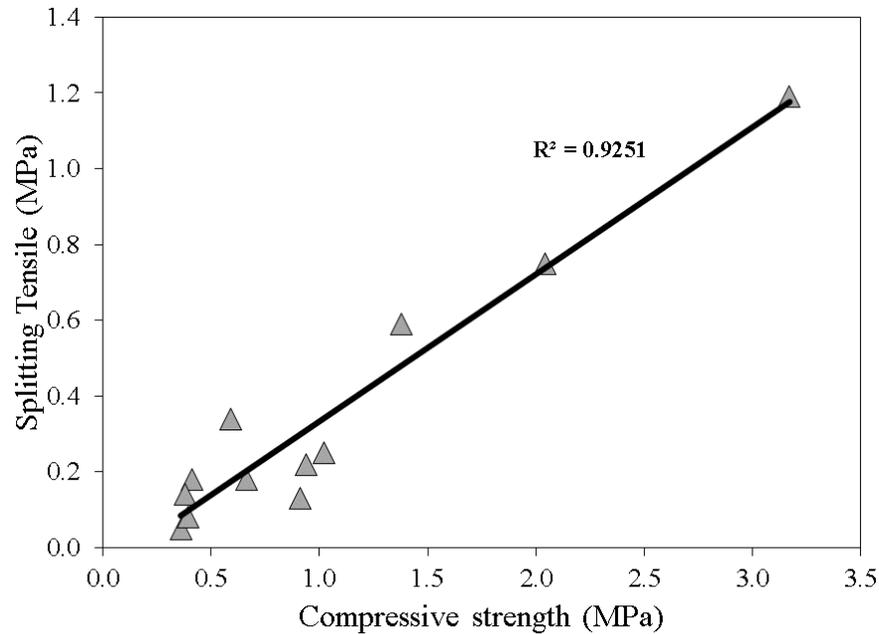


Figure 4.7 Relationship between splitting tensile strength and achieved compressive strength for all CLSM mixtures.

4.3.6 Absorption rate

An absorption test was carried out at the age of 28 days, and the results are shown in **Fig. 7**. The hygroscopy is the phenomenon of attracting and holding water molecules via either absorption was 10.16 to 14.52% for mixtures with 90 kg/m³ cement and various amount of agro-waste. Generally, increasing agro-waste content resulted in higher absorption. For instance, adding 10%, agro-waste increased the absorption rate with about 21.26% than that of the control mixture without agro-waste. This can be attributed to the high-water absorption rate for the agro-waste as compared with sand. Similar trends were found for other mixtures with 30 kg/m³ and 60 kg/m³ cement content. The lower the cement content, the higher the absorption rate (**Fig. 8**). For instance, G30 mixtures showed a 19% higher absorption rate than that of G90 mixtures. This can be ascribed to the low hydration product formation due to the lower cement content and, consequently, lower pozzolanic reactions. This resulted in an open microstructure, as confirmed by the porosity results shown in **Figure 4.8**. The porosity of the CLSM specimen with 20% agro-waste was about 29.83% for 30 kg/m³ mixtures compared to around 25.27% for 90 kg/m³ mixtures. Moreover, the porosity increased as the quantity of agro-waste increased (**Figure 4.7**). This can be ascribed to the low volume to the density ratio of agro-waste compared to sand. Replacing the same volume of sand

with agro-waste, which has a lighter weight, decreases the volume density for CLSM [141]. Consequently, a higher amount of internal pores will need to be filled with hydration products. Therefore, the efficiency of the filling effect of fly ash and pozzolanic reaction will decrease, leading to the porosity of the CLSM.

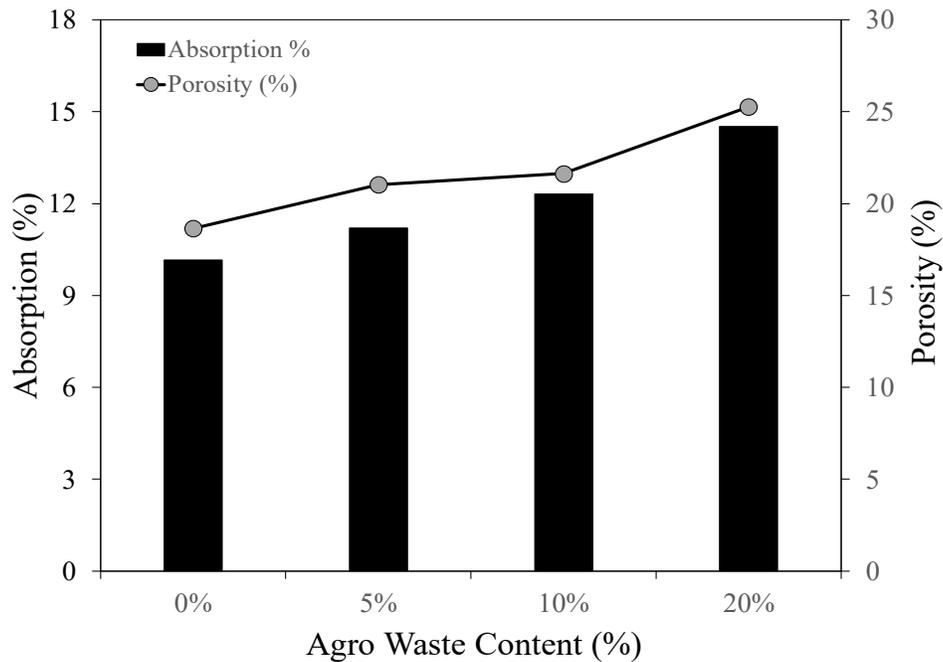


Figure 4.8 Absorption and porosity for 90 kg/m³ CLSM mixture with various agro-waste contents

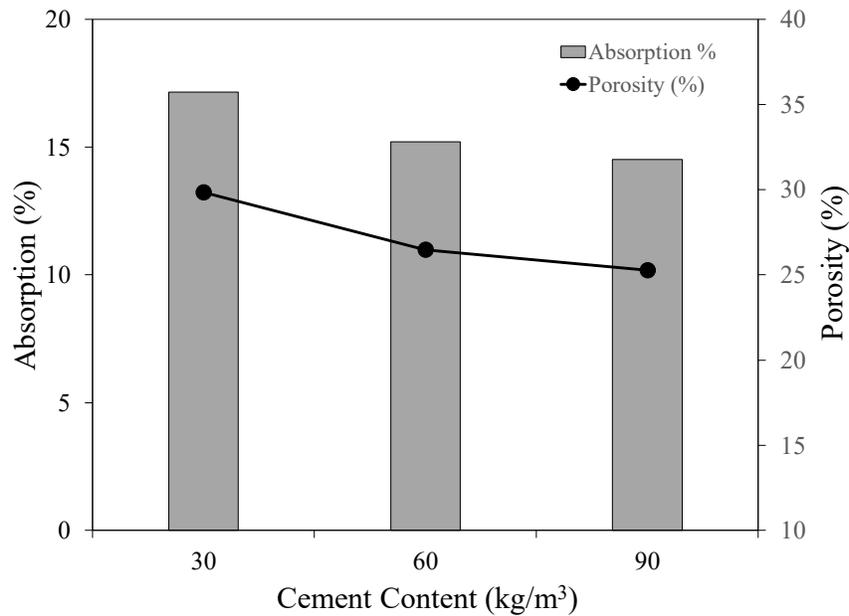


Figure 4.9 Absorption and porosity for CLSM mixture with 20% agro-waste and different cement contents

4.3.7 Ultrasonic pulse velocity

Figure 4.9 summarizes the ultrasonic wave velocities for all tested mixtures. General, the measured wave velocity increased with time. For instance, for G90 mixtures, the wave velocity at age 28 days was about 24.76% higher than that at age seven days. This can be attributed to the progress of hydration and the formation of different products that densify the microstructure. This agrees with the development of compressive strength[197]. **Figure 4.10** shows a good correlation between the achieved compressive strength and measured wave velocity. Similar to compressive strength, the lower the cement content, the lower the measured UPV, which can also attribute to the lower degree of hydration. Fly ash is known to have a prolonged rate of hydration. Moreover, the pozzolanic reaction of fly ash will mainly depend on the amount of calcium hydroxide (CH) provided by cement. Calcium hydroxide will react with silicate from fly ash to form more calcium silicate hydrate (CSH), which is the binding material and mainly responsible for strength gain. Hence, for lean mixtures with low cementitious content, lower CSH will be formed and, consequently, lower density microstructure. Therefore, the measured ultrasonic pulse velocity will decrease. Also, the high porosity of the agro-waste itself will decrease the wave velocity[198]. For

instance, increasing the agro-waste content from 5% to 20% for mixtures with 90 kg/m³ results in around 28.54% reduction in the measured wave velocity.

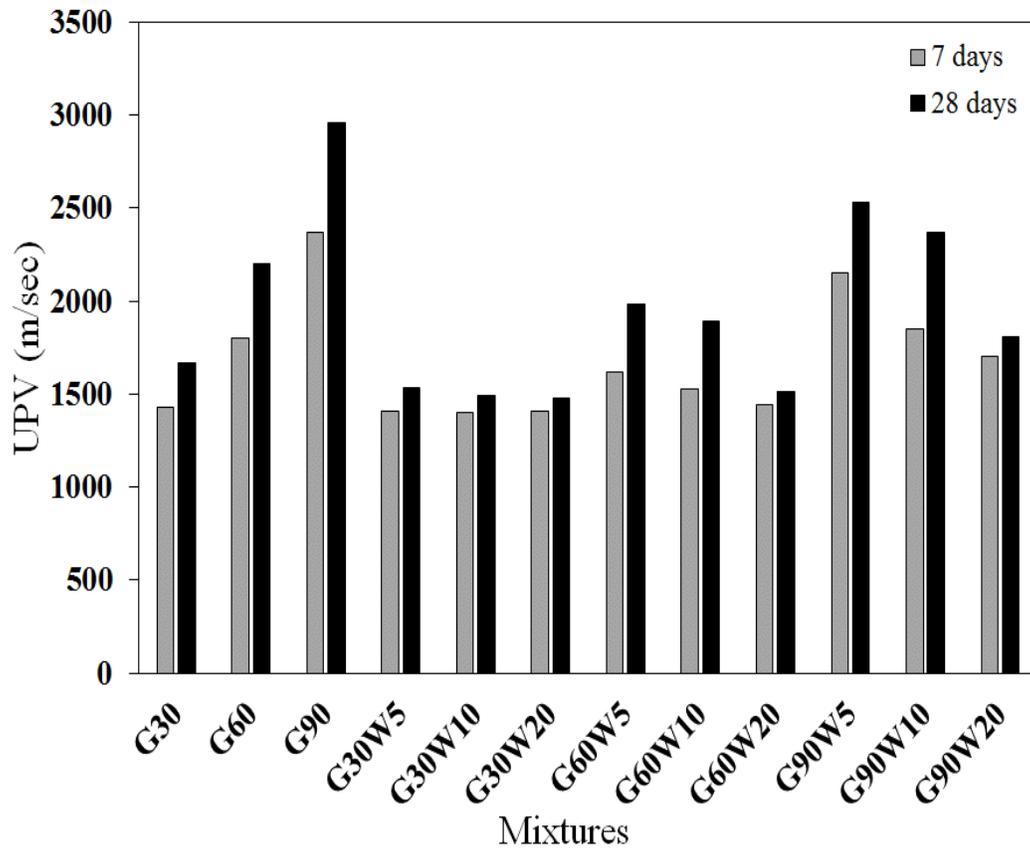


Figure 4.10 Ultrasonic pulse velocity for all tested CLSM mixtures at different ages

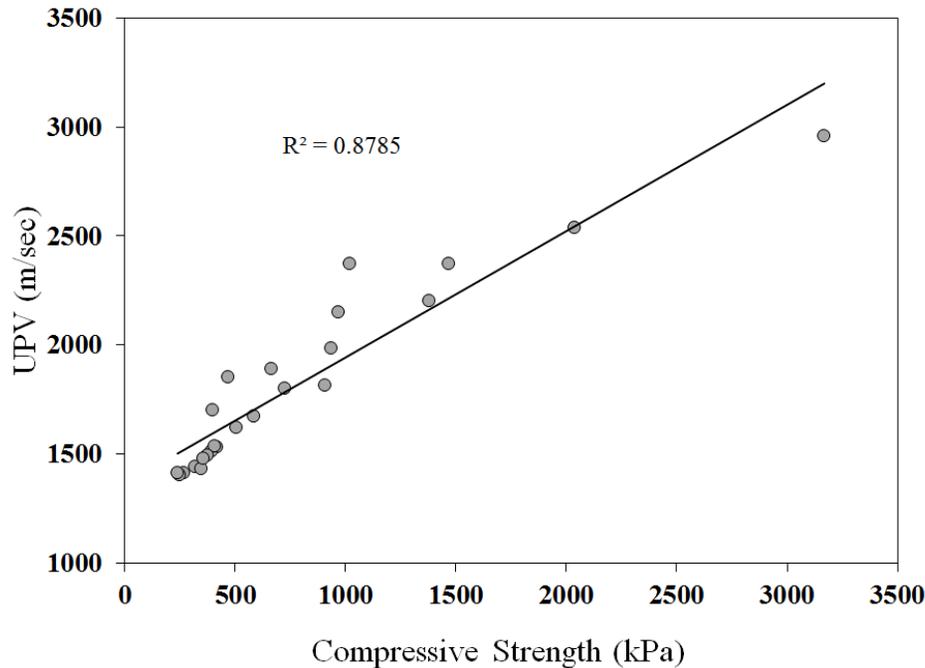


Figure 4.11 Relationship between measured Ultrasonic pulse velocity and achieved compressive strength for all CLSM mixtures.

4.3.8 Shrinkage

The shrinkage for CLSM mixtures represented by the change in the length of the prismatic specimens over the investigated period shown in **Figure 4.11**. It shows mentioned that shrinkage for mixtures with 30 kg/m^3 and 60 kg/m^3 were not evaluated as the specimens were too weak and easily broken. Mixtures G90 and G90W05 exhibited shrinkage within the standard ultimate shrinkage limit for CLSM (i.e. 0.02% to 0.05%), according to [186]. Conversely, shrinkage for G90W10 and G90W20 mixtures exceeded the limits for CLSM (i.e. 0.05%); however, it was still below the ultimate shrinkage for concrete (i.e. 0.1%)[131]. This behaviour is related to several factors, including water content, bleeding, and the nature of used agro-waste. To meet the flowability requirements, high water to powder ratio in the range of 1 to 1.5 used. This high amount of water content is expected to increase the amount of evaporable water and, consequently, the expected shrinkage. Moreover, mixtures with high bleeding values (G90 and G90W05) exhibited lower shrinkage as the water dried from the surface of the material rather than from the bulk[194]. Furthermore, restraining materials (i.e., aggregate) in cementitious materials has a direct effect on the shrinkage[199]. Hence, replacing sand with high stiffness with agro-waste with

a low stiffness will reduce the restraining effect of the aggregate resulting in a higher shrinkage. The higher the replacement ratio, the higher the shrinkage [141].

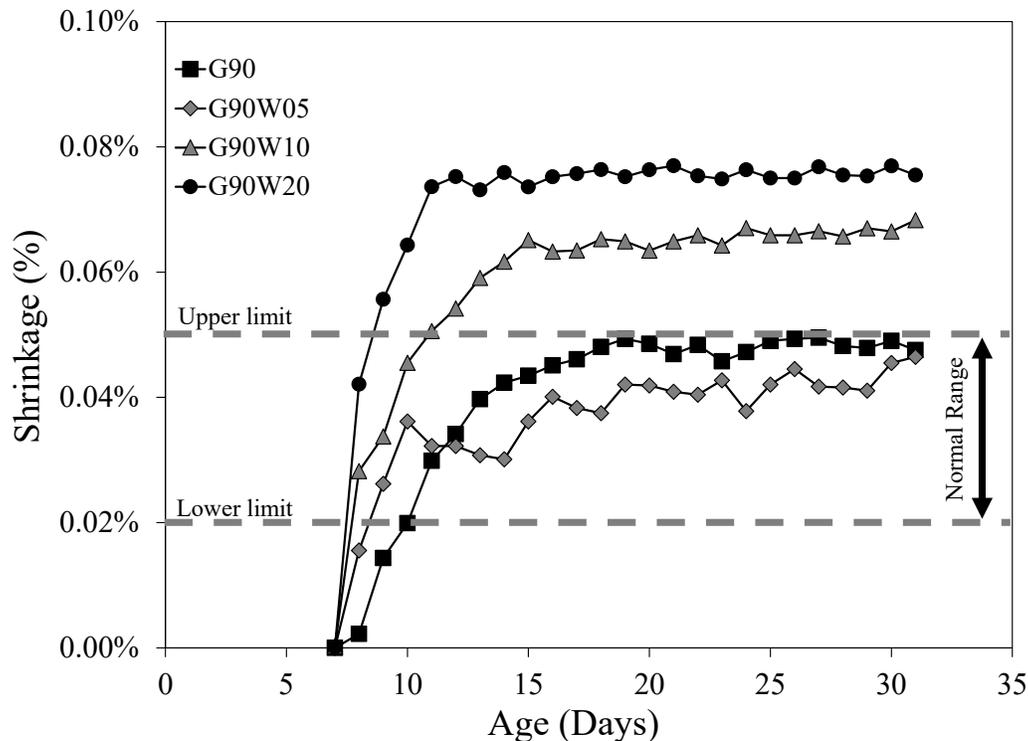


Figure 4.12 Shrinkage development for 90 kg/ m³ CLSM mixtures.

4.3.9 Sulphate attack

The performance of CLSM mixtures with and without agro-waste under Sulphate exposure were evaluated based on the number of cycles in Sulphate solution, mass loss, and residual strength. Figure 4.13 shows the number of cycles sustained by CLSM specimens soaked in the Sulphate solution. The trend is not the same for all mixtures. For mixtures with a high cement content (i.e., 90 kg/m³), the higher the agro-waste content, the lower the number of sustained cycles. For instance, 90 kg/m³ cement mixtures with agro-waste contents 5% and 20% sustained 15 and 11 cycles before failure, respectively. On the other hand, mixtures with low cement (i.e. 30 kg/m³), adding agro-waste up to 10%, had an increased number of sustained cycles, while a significant reduction reported at higher contents (i.e. 20%). For moderate cement mixtures (i.e. 60 kg/m³), no clear trend could be identified; however, at 20% agro-waste content, the specimens could not sustain more than ten cycles. Generally, all CLSM specimens showed a mass loss after soaking in the sulphate. The higher the number of cycles, the greater the mass loss. The mass loss varied

based on the cement content. For instance, mixtures with 90 kg/m^3 cement, the mass loss was in the range of 2.59% to 7.19%, while for 30 kg/m^3 cement mixture, the range was 10.13% to 20.81% depending on the number of cycles.

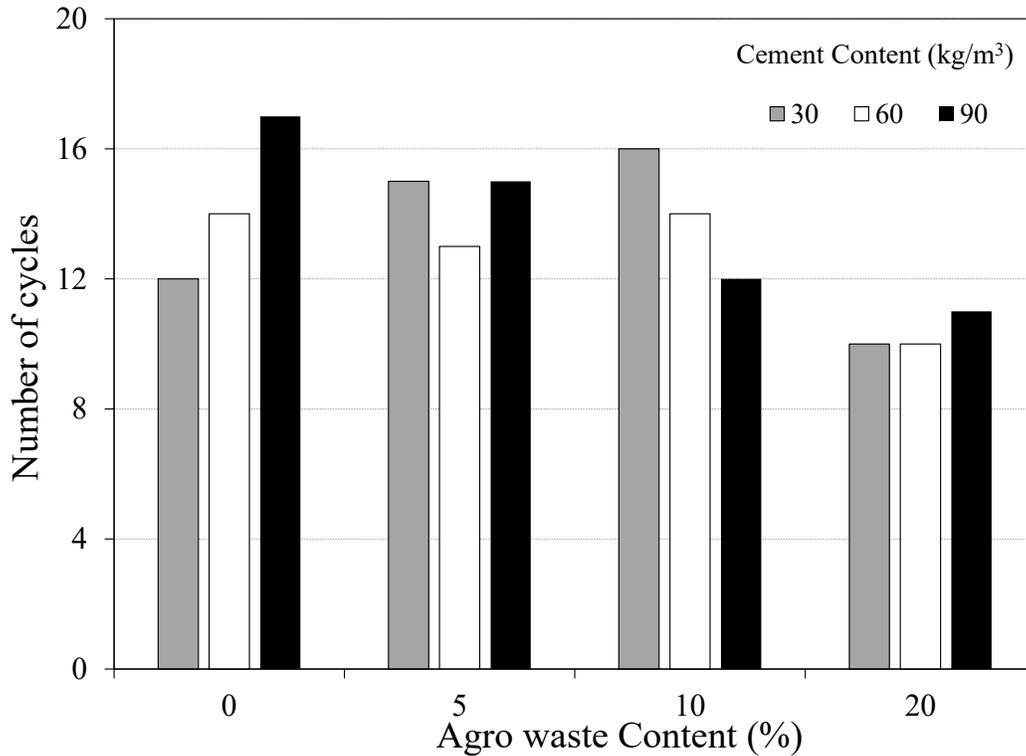
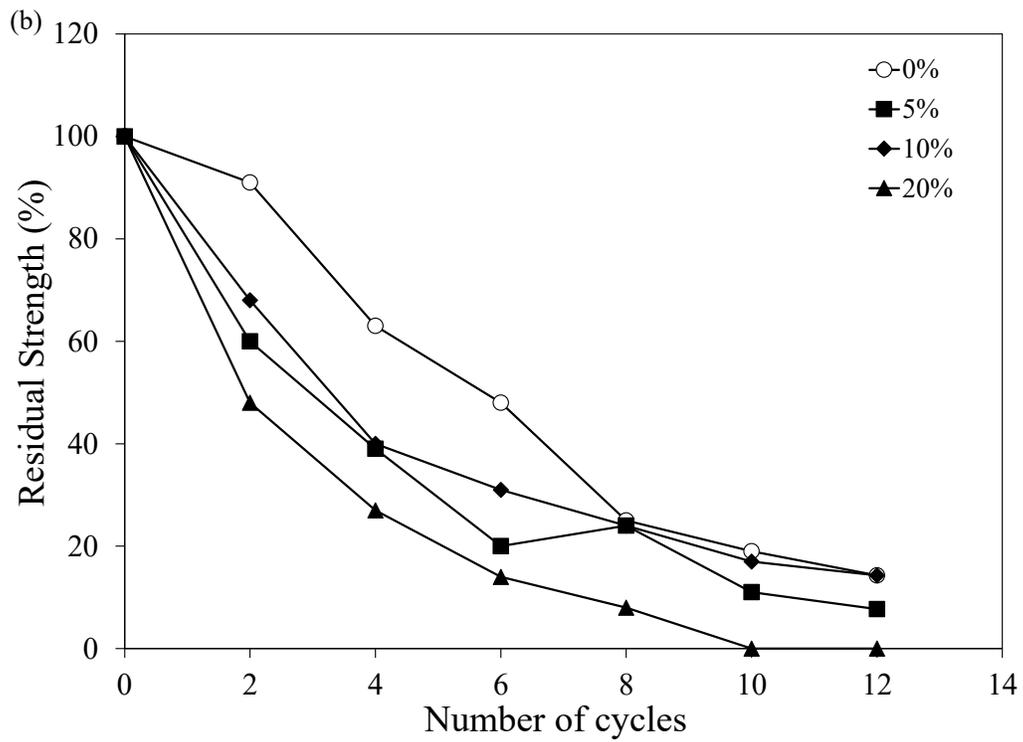
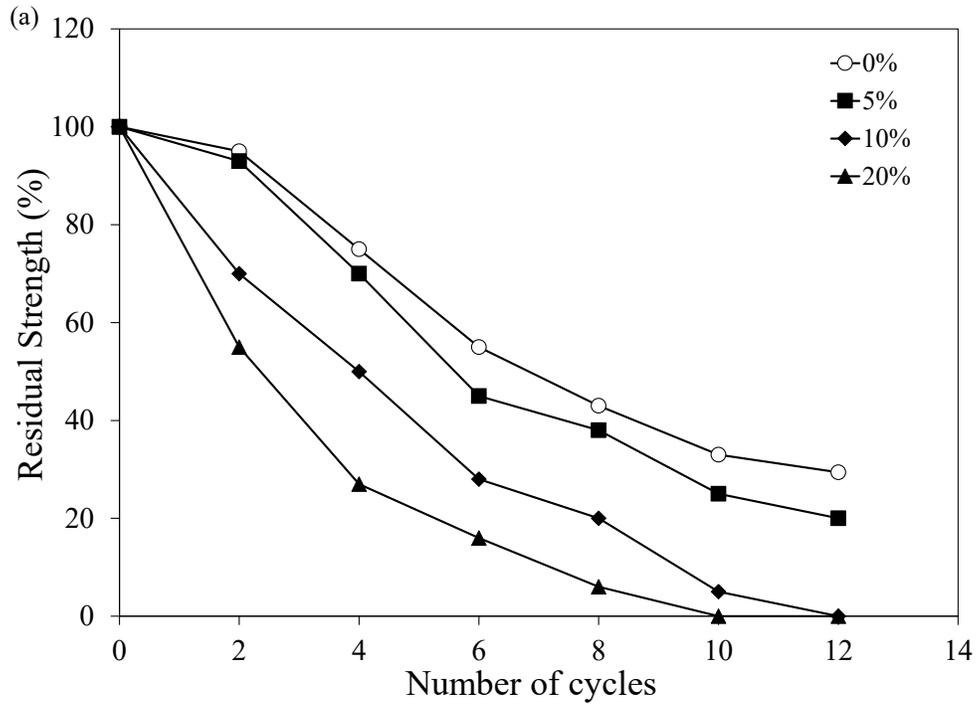


Figure 4.13 Numbers of sustained cycles in sulphate solution for all CLSM mixtures

Figure 4.14 shows the residual strength for CLSM specimens up to 12 cycles in sulphate solutions. Generally, the strength of CLSM specimens reduced with the increase of the number of cycles regardless of the cement and agro-waste contents. For instance, for 5% agro-waste content, mixtures with 60 kg/m^3 and 90 kg/m^3 cement showed residual strength of 39% and 70% after 4 cycles and 11% and 25% after 10 cycles, respectively. Also, the reduction in strength was more severe during the initial cycles for lean mixtures compared with that for stronger mixtures. For example, at 20% agro-waste, mixtures with 30 kg/m^3 cement lost around 60% of its 28 days strength after two cycles, while mixtures with 90 kg/m^3 lost about 42% under the same condition.



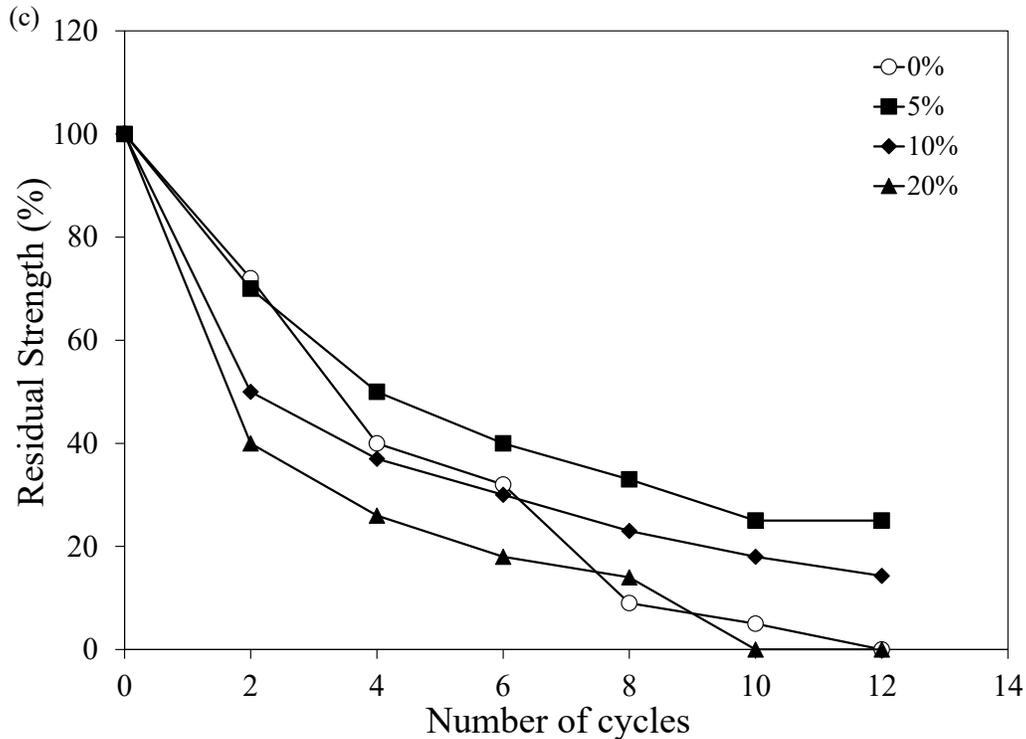


Figure 4.14 Contd': Residual strength for CLSM mixtures incorporating different agro-waste content and cement content of a) 90 kg/m³, b) 60 kg/m³, and c) 30 kg/m³.

This unclear trend with varying cement and agro-waste contents can attribute to the effects of competing factors, which may oppose or comply with each other. Generally, adding agro-waste had increased the Sulphate attack as indicated by residual strength results. The CLSM is a porous cementitious material facilitating the ingress of Sulphate salts carried by water. This highly acidic environment is likely to react with agro-waste and hydrolysis, such as waste[141]. The change in the solution colour confirmed this after submerging the CLSMs specimens.

Moreover, in such a low pH environment (i.e., < 10), ettringite is unstable; hence, massive expansion due to its formation is not anticipated[200]. Moreover, the high fly ash content will also contribute to reducing the pH for the pore solution by consuming the calcium hydroxide in the pozzolanic reaction, which will be promoted by the high temperature during the drying period [201]. Simultaneously, the pozzolanic reaction will enhance the strength growth, which may contribute to the residual strength.

Another parameter that is anticipated to contribute to the degradation of CLSM specimens is the cyclic dry and wet stages in the sodium Sulphate solution. Exposure to repetitive wet and dry periods leads to volume instability (i.e., dry shrinkage and wet expansion deformation). This deformation will induce internal stresses leading to microcracking once exceeding the tensile strength for the CLSM mixtures. The formed cracks would accelerate shrinkage and swelling deformation, resulting in further propagation for the cracks, breakage for the internal structure, and, finally, failure for specimens[202].

4.4 CONCLUSION

The fresh and hardened properties of CLSMs using agro-waste, along with durability performance, were investigated. This research focus was to examine the potential of using agro-waste to produce CLSM mixtures that satisfying workability and performance requirements. The following conclusions can be drawn:

1. All mixtures with agro-waste displayed excellent flow properties. The water demand of a CLSM mixture increases as agro-waste content increased. Hence, the flowability and filling capacity of CLSM mixtures decrease as agro-waste content increased at constant water content.
2. Increasing agro-waste content has the beneficial effect of reducing bleeding of CLSM mixtures
3. Agro-waste CLSM mixtures are easily excavatable with removability modulus less than one except for mixtures with 90 kg/m^3 cement. This indicates that these high cement content CLSM can accommodate a higher amount of agro-waste (i.e., > 20%).
4. The study confirms that CLSMs with high agro-waste content can be developed with acceptable properties and without affecting the correlation between them. Correlation between agro-waste CLSMs compressive and tensile strengths was similar to that of conventional CLSMs without agro-waste.
5. High agro-waste content reduces the sulphate resistance of CLSM specimens. The optimum agro-waste content to have adequate sulphate resistance will vary depending on the cement content. Cyclic wet and dry in sodium sulphate solution escalate the effect of volume instability.

CHAPTER 5. PROPERTIES OF ALKALI ACTIVATED BIO-BASED CONTROLLED LOW STRENGTH MATERIAL

Alkali activated materials (AAM) have proven to be a potential green sustainable alternative binder for cement in many construction applications. Hence, this study investigates the feasibility of producing green bio-based construction materials, specifically controlled low-strength material (CLSM). Alkali activated CLSM mixtures incorporating various contents of agriculture residuals were produced. Besides, a CLSM mixture with ordinary Portland cement (OPC) was also prepared for comparison. Fresh and hardened properties for all mixtures, including flowability, bleeding, unit weight, strength, were evaluated. In addition, ultrasonic pulse velocity and sulphate attack resistance were evaluated for various CLSM mixtures. Generally, increasing agriculture residuals contents reduced the fresh and hardened properties and sulphate attack resistance. Simple fine-tuning for the mixture design will successfully offset these adverse effects. The reduction in mechanical properties was within the acceptable limits for CLSM. Sodium hydroxide activator is suitable for the low strength requirements and easy excavability for CLSM. The findings of this study will pave the way for wider implementation of agriculture residuals in the construction sector, increasing sustainability.

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5.1. INTRODUCTION

Construction material manufacture consumes a significant amount of natural resources, which has a serious ecological impact. Moreover, concrete is the most used construction material for many applications. This huge demand is expected to result in approximately 18 billion tons of concrete by 2050 [147, 177]. Since a significant volume of concrete, about 60 to 70%, is natural aggregates, this significant increase in concrete demand will have serious environmental and ecological implications. Therefore, the use of various eco-friendly alternative aggregates has been proposed by several researchers. For instance, waste tires, industrial wastes, and agricultural wastes have been used as alternatives for natural aggregates in the concrete industry [3-6]. In particular, in the context of green and sustainable buildings, using agricultural waste/residuals as a replacement to natural aggregate has attracted the attention of many researchers. Agricultural waste/residuals are eco-friendly, low carbon, and sustainable materials [2, 62, 178-183]. Moreover, these wastes/residuals are available with a wide variety of shapes, sizes, and types, which increase their potential to be implemented in many applications [2]. Several agricultural wastes/residuals such as bamboo, coconut shells, and others were applied successfully in structural and non-structural cementitious construction materials [182, 184, 185].

One of the promising applications for agricultural wastes/residuals is controlled low strength materials (CLSM), thanks to its low strength requirements (around 8.3 MPa or less), according to ACI 229R-13. CLSM is also known as soil-cement slurry, flowable filler or mortar, structural backfill, and plastic soil-cement [131, 141, 186]. The low targeted strength of CLSM facilitated the incorporation of a high amount of wastes such as treated oil sands waste or chip ballast as a partial replacement of fine aggregate [131, 188].

On the other hand, the ACI 229R-13 report for CLSM shows that cement and fly ash (FA) is the main composition for the binding material; however, FA content can reach up to 85% of the total binder. The low cement content, compared to conventional concrete, increased CLSM sustainability [8, 203]. However, even with such low content, carbon dioxide emission by cement cannot be ignored, as on a larger scale, its harmful environmental impact will be significant [8, 204, 205]. Therefore, using a more environmentally friendly binder as an alternative for cement will increase CLSM sustainability. Among available alternative binders for cement, alkali-activated materials (AAMs) is one of the most promising binders.

In AAMs, aluminosilicate materials (so-called precursors) are activated by high alkalinity solution (so-called activator). The highly alkaline conditions rapidly dissolve and free $[\text{SiO}_4^{4-}]$ and $[\text{AlO}_4^{4-}]$ tetrahedral units from the reactive aluminosilicates and release them into the solution. The tetrahedral units are alternatively linked to polymeric precursor by sharing oxygen atom, forming polymeric Si–O–Al–O bonds. This process is known as polymerization [206, 207]. Interestingly, FA is an aluminosilicate material and had been used as solid raw material for AAM. Various alkaline activators such as sodium hydroxide (NaOH), potassium hydroxide (KOH), sodium silicate (Na_2SiO_3), and potassium silicate (K_2SiO_3) have been used to activate fly ash AAMs. The produced AAMs are characterized by low CO_2 emission, superior properties including high chemical resistance, outstanding mechanical properties, and durability performance compared to that of the OPC. Hence, AAMs are a promising alternative binder for OPC to produce building materials more synchronous with nature [137, 203, 208].

In this study, the feasibility of producing bio-based green CLSM by combining AAMs and agriculture residuals was examined. The successful combination of agriculture residuals and AAMs will benefit both the construction and agricultural sectors through saving energy, natural resources, landfill space, and disposal cost.

5.1 EXPERIMENTAL WORK

5.1.1 Material

Class-F fly ash (FA), according to ASTM C618, was used as the main precursor materials for all tested CLSM mixtures. In addition, granulated blast furnace slag (hereafter referred to as slag) with an average particle size value around $14.5 \mu\text{m}$ was also used to enhance early strength gaining. The basicity coefficient $[K_b = (\text{CaO} + \text{MgO}) / (\text{SiO}_2 + \text{Al}_2\text{O}_3)]$ for the used slag was 1.06.

Table5-1 shows the chemical and physical properties for fly ash and slag. Sodium hydroxide (NaOH) was used as an activator. Sodium hydroxide solution was prepared by dissolving 99% pure NaOH flakes in distilled water. For cement-based mixtures, General used ordinary Portland cement (OPC) according to the CSA-3001-03 with Blaine fineness of $360 \text{ m}^2/\text{kg}$, and a specific gravity of 3.15 was used. Natural river sand with particle size distribution complies with ASTM C33 [26] for fine aggregate, and a specific gravity of 2.65 was used. The waste (i.e. will be named so after “agro-waste”) was a fine black spruce residual. It had a micro-size irregular particle

mixed with few micro size fibrous shape particles with a specific gravity 0.41. The black spruce grows in a broad transcontinental band from Newfoundland (Canada) to Alaska (United States) [189]. It accounts for around 12% of the total Canadian softwood inventory. However, it was implemented in different applications; disposal of remaining black spruce residuals is one of the main challenges facing the forest industry [190]. In this study, the OPC-CLSM mixture composition was selected based on the proportion guidelines reported by ACI committee 229 [186]. The mixture composition for the AAM-CLSM control mixture without agro-waste was adjusted by trial-and-error to achieve the flowability range for OPC-CLSM. Then, the AAM-CLSM control mixture was modified by the incorporation of agro-waste as partial replacement of sand by volume at rates of 5%, 10%, 20%, 30%, and 40%. Mixture proportions are shown in **Table 5-2**

Table 5-1 Chemical and physical properties of OPC, fly ash, and Slag

		OPC	Fly Ash	Slag
SiO ₂	(%)	19.80	43.39	36.50
Al ₂ O ₃	(%)	4.90	22.08	10.20
CaO	(%)	62.30	15.63	37.60
Fe ₂ O ₃	(%)	2.30	7.74	0.50
SO ₃	(%)	3.70	1.72	3.10
K ₂ O	(%)	0.83	2.40	0.40
Na ₂ O	(%)	0.34	1.01	0.30
MgO	(%)	2.80	--	11.80
TiO ₂	(%)	0.11	--	1.00
Loss on ignition	(%)	1.90	0.58	0.58
Specific gravity	--	3.15	2.50	2.92
Surface are	(m ² /kg)	360	280	515

Table 5-2 Mixture compositions for tested CLSM mixtures

Mixture	Cement (kg/m ³)	Fly Ash (kg/m ³)	Slag (kg/m ³)	Sand (kg/ m ³)	Water (kg/ m ³)	Waste (% by vol.)	NaOH (kg/ m ³)
CC	90	148	---	1655	297	---	--
CA	--	190	50	1655	276	0.0	57
M05	--	190	50	1572	278	5.0	58
M10	--	190	50	1490	287	10.0	59
M20	--	190	50	1325	303	20.0	63
M30	--	190	50	1160	348	30.0	73
M40	--	190	50	995	370	40.0	77

5.1.2 Mixing procedure

The following mixing procedure was followed according to previous studies [131, 191]. Dry solids components (fly ash, slag, agro-waste, and sand) were first mixed for one minute to ensure uniform distribution. Half of the sodium hydroxide (NaOH) solution was added gradually while continue mixing for one more minute. The mixture was allowed to rest for 1 min, then the rest of the solution was added to the mixture and continue mixing for an additional 2 minutes. For all tested mixtures, additional water was added while continuously mentoring the flowability targeting a normal flowability in the range of 150 mm to 200 mm as recommended by [186]. The amount of NaOH was adjusted to have the same molarity for all mixtures. For OPC-CLSM, the same procedure was followed, expect no slag was added, and normal tape water was used for mixing.

5.2 Testing

A series of fresh and hardened tests were conducted to evaluate various properties of alkali-activated Bio-based CLSM. Fresh properties, including flowability, bleeding, and unit weight was evaluated following ASTM standards D6103-04 (Flow Consistency of Controlled Low Strength Material), ASTM test method C232 (Standard Test Method for Bleeding of Concrete), and ASTM D6023-07 (Density, Yield, Cement Content, and Air Content (Gravimetric) of CLSM) respectively. Cubic specimens 50 × 50 × 50 mm were used to evaluate the compressive strength at ages 7, 14, and 28 days using a strain-controlled unconfined compressive strength machine and flowing the same loading rate recommended by ASTM test method D4832-10 (Standard Test

Method for Preparation and Testing of CLSM Test Cylinders). Specimens were cured at curing room (temperature $22 \pm 2^\circ\text{C}$ and relative humidity $95\% \pm 3\%$) inside the mold uncovered until the testing age due to the insufficient early strength of CLSM mixtures. Moreover, splitting tensile strength was conducted at the age of 28 days following ASTM standards C496/C496M (Standard Test Method for Splitting Tensile Strength of Cylindrical Concrete Specimens). The ultrasonic pulse velocity test examined variations in alkali-activated bio-CLSM density along with detecting any internal defects according to ASTM C597 (Standard Test Method for Pulse Velocity Through Concrete). Water absorption and pores conductivity were evaluated following ASTM C642 (Standard Test Method for Density, Absorption, and Voids in Hardened Concrete). Sulphate attack resistance was evaluated following the same procedure conducted by [141]. Total porosity was evaluated for selected samples using Mercury Intrusion Porosimetry. Specimens were exposed to dry/wetting cycles at age 28 days. For one complete cycle, specimens were dried in an oven for one day at $100 \pm 5^\circ\text{C}$ and then soaked for a day in a highly concentrated sodium sulphate solution at 22°C . The solution was prepared by dissolving 350 g of the anhydrous sodium sulphate per 1 L of water, according to [141]. Changes in residual strength for specimens were monitored with the number of cycles.

5.3 RESULTS AND DISCUSSION

5.3.1 Flowability

All flowability results for tested CLSM mixtures are illustrated in **Figure 5.1**. As previously mentioned, the amount of added water was adjusted to achieve an adequate flowability within the range of 150-200 mm according to the ACI 299 [186]. Alkali-activated CLSM mixtures without agro-waste exhibited higher flowability than that of cement-based CLSM. This can be attributed mainly to the increase in FA content, which had spherical particle shape (**Figure 5.2**), facilitating particle movement and reducing the friction[209]. All mixtures exhibited normal CLSM flowability within the range of 155 mm to 195 mm. Generally, increasing the agro-waste content reduced flowability, which requires increasing the amount of water to achieve the targeted flowability. This was clear in mixtures incorporating high agro-waste contents (i.e. >10%). Adding the fine agro-waste (which is finer than sand) as a replacement of sand has several effects. The addition of such small particles materials will fill the voids between large particles freeing entrapped water, which will enhance flowability [131]. Conversely, this fine material will increase

surface area significantly, leading to higher water adsorbed to the particle surface, leading to lower workability. Besides, agro-waste itself will absorb water (i.e. agro-waste absorption is 4.5%), which will reduce the amount of free water [23]. It seems that up to 10% agro-waste addition, there was an acceptance for the performance as all mixtures flowability was within the desired flowability range for normal flow CLSM. Hence, almost the same water-to-solid ratio was used. At higher agro-waste contents, additional water was necessary to maintain the targeted flowability range. This indicated that at such high contents (i.e. $\geq 20\%$), the increase in the surface area and water absorption were dominating the flowability behaviour over the filling effect.

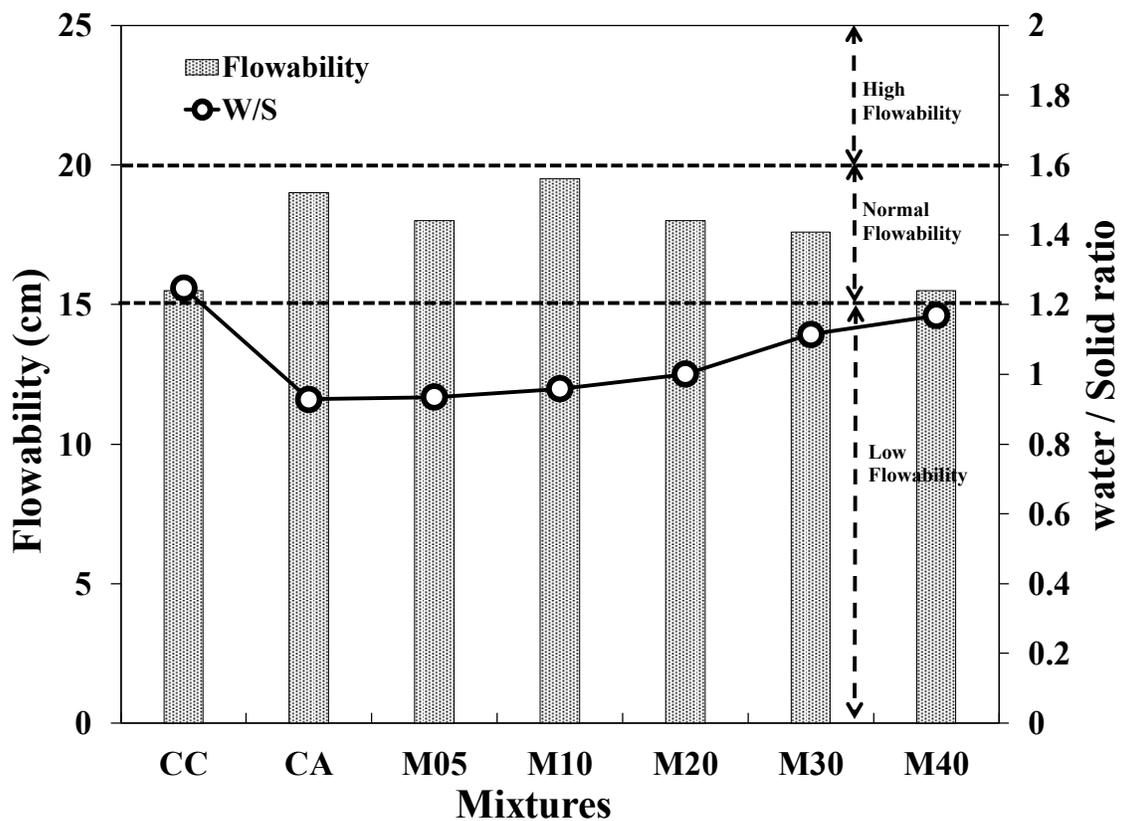


Figure 5.1 Flowability for all tested CLSM mixtures

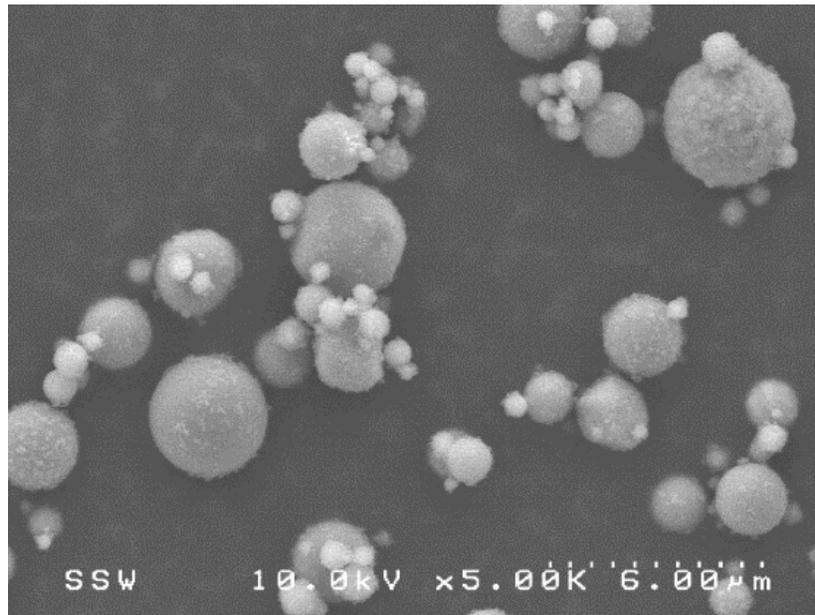


Figure 5.2 spherical particle shape for the used FA.

5.3.2. Bleeding

According to previous work [193], a maximum of 5% bleeding is allowed for a stable CLSM mixture. Bleeding values for all mixtures did not exceed the maximum limit, as shown in **Figure 5.3**. Alkali activated materials without agro-waste showed higher bleeding than that of the OPC-CLSM mixtures. Adding fly ash is expected to reduce bleeding as the increase in flowability, induced by fly ash addition, will allow the reduction of water content [209, 210]. However, this was not the case for AAM-CLSM. The increase in flowability induced by increasing FA content was not enough, and more water was to achieve the targeted flowability range. Hence, it seems that the used amount of water to achieve flowability was high; however, it was accepted as the flowability and strength requirements were fulfilled. It is anticipated that if the water for AAM-CLSM were reduced to achieve similar flowability for that of the cement-based CLSM, the bleeding would be less.

On the other hand, increasing the amount of agro-waste had reduced the bleeding value. For instance, adding 10% agro-waste as a replacement of sand had decreased the bleeding value by about 40%. This can be attributed to the high water absorption agro-waste (i.e. 4.5%) [23]. Besides

the water absorbed, the finer size for the agro-waste compared to sand will increase the water demand to cover its surface. As a result, free water in the mixture for bleeding during the setting period will decrease significantly [194]. One interesting point, for mixtures incorporating high agro-waste (i.e. > 20 %), the bleeding was null even after increasing the water content. This confirms the flowability results indicating the scarcity of water. The high absorption of agro-waste, along with the increase in the surface area due to agro-waste addition, was high enough to consume all additional free water.

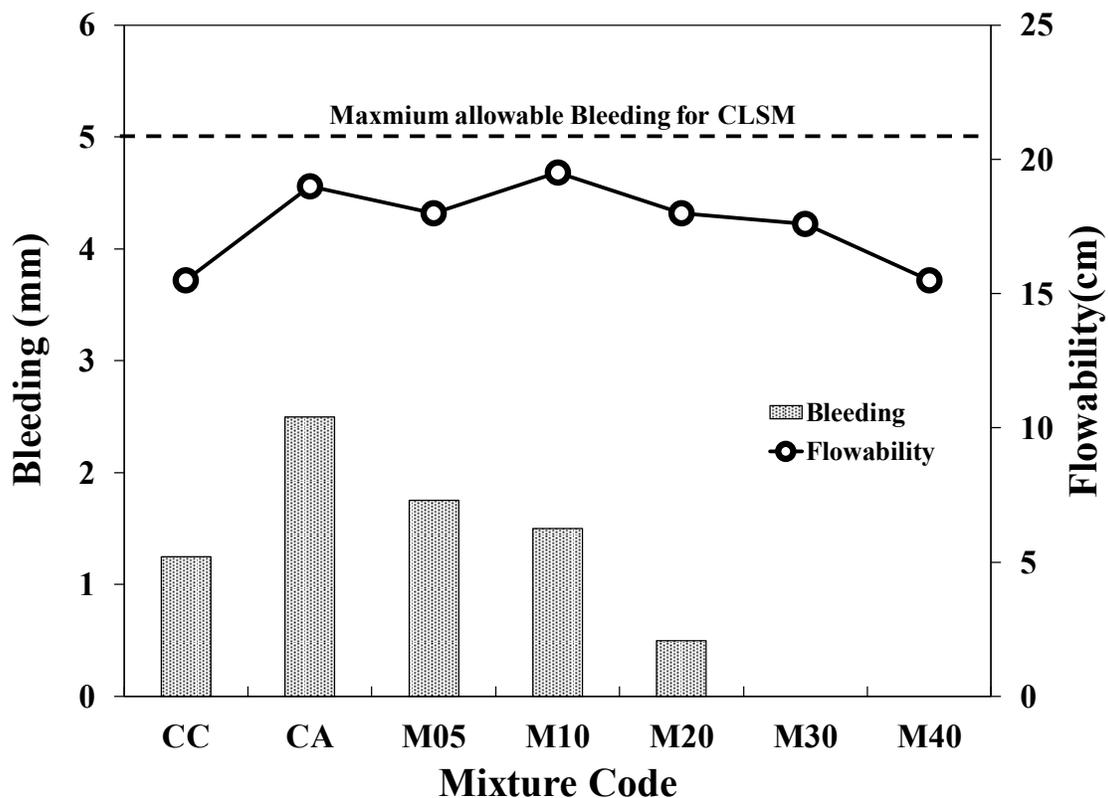


Figure 5.3 Bleeding for all tested CLSM mixtures

5.3.2 Density

Changes in the wet and dry densities for various CLSM mixtures as a result of adding agro-waste is illustrated in **Figure 5.4**. Interestingly, the wet density of the AAM-CLSM mixture without agro-waste was almost the same as that of the cement-based CLSM. This can be ascribed to the differences in the mixtures composition and the specific weight for used materials. For instance, fly ash has lower specific gravity than cement, while the mixing water was pure in cement-based CLSM compared to sodium hydroxide solution in AAM-CLSM.

On the other hand, the higher the added amount of agro-waste, the lower the wet densities for the AAM-CLSM mixtures. Wet densities of the AAM-CLSM materials with and without agro-waste ranged from 1777 kg/m³ to 2228 kg/m³. These values were within the normal fresh density range for CLSM (1840 to 2320 kg/m³) according to ACI Committee 229 report, except for M40, which can be considered as low-density CLSM. Moreover, the maximum variation in wet densities was only about 6%. This can be directly related to two compensating factors: 1) Reduction due to the low density of agro-waste compared to sand and 2) Increase due to adding more water to achieve required flowability. Generally, adding agro-waste will reduce density. Conversely, adding more water to adjust flowability (i.e. due to water absorption by agro-waste) will increase the water content, and consequently, the wet density.

Dry densities for AAM-CLSM was substantially less than fresh values considering the high water loss. In fact, all AAM-CLSM with agro-waste above 5% (i.e. $\geq 10\%$) is meeting the low-density CLSM fresh density range (320 to 1920 kg/m³) according to ACI Committee 229 report (**Figure 5.4**). This illustrates the potential of producing low-density AAM-Bio-Based CLSM for various construction applications at which low dead load is a critical requirement.

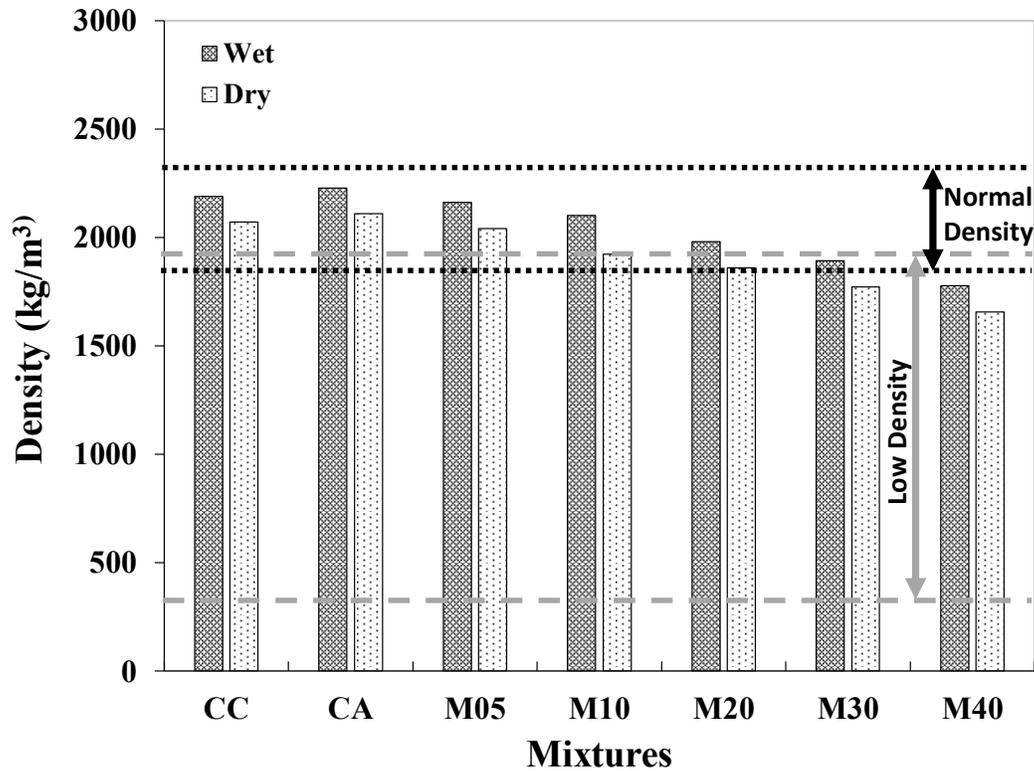


Figure 5.4 Wet and Dry densities for CLSM mixtures

5.3.3 Compressive Strength

Generally, the compressive strength of cement-based CLSM is controlled by binder content (i.e. cement and fly ash), mixing water and fine aggregate[211]. Cement added to the mixture is typically the primary factor controlling the strength of the specimens. The formed calcium hydroxide (CH) will be consumed in the pozzolanic reaction of fly ash, forming more calcium silicate hydrated (CSH) (i.e. which is responsible for strength development) [195]. This aligns with the development of cement-based CLSM shown in **Figure 5.5**. However, for alkali-activated systems, the slag-to-fly ash ratio, the molarity for NaOH solution, and the water to binder ratio are used to control the compressive strength development[19]. Fly ash and slag will be chemically activated by the addition of sodium hydroxide solution forming hydrated calcium aluminates and aluminosilicates (C-A-H and C-A-S-H phases) [212].

In the present study, the slag-to-fly ash ratio and the molarity for NaOH solution were maintained the same for all mixtures. The amount of added agro-waste and corresponding water content to achieve the targeted flowability were utilized to control the AAM-CLSM strength. In other words, maximizing the amount of added agro-waste while meeting the strength requirements for CLSM.

As shown in **Figure 5.5**, AAM-CLSM had a high compressive strength than that of the OPC-CLSM. This is agreed with previous studies showing that AAM has a higher strength than that of cement [213, 214]. At ambient temperature, the aluminosilicate phases of fly ash and slag undergo polymerization process when activated by the NaOH solution. The added slag will rapidly react with NaOH solution than the fly ash releasing calcium (Ca^{2+}), Silica (Si^{4+}), and Aluminum (Al^{3+}) ions in the aqueous solution. Moreover, the slag will undergo hydration reaction due to its higher calcium content besides the geopolymerization reaction. Hence, hydration and geopolymerization reaction products will form simultaneously, leading to a higher strength gain [215].

AAM-CLSM mixtures did not exceed the upper limit for CLSM (i.e. 8.3 MPa) for applications where future excavation is not desired (i.e. structural filling). Generally, the strength for all AAM-CLSM increased with time regardless of the amount of added agro-waste. The higher the added amount of agro-waste, the higher the reduction in strength. For instance, increasing the agro-waste content from 10% to 30% resulted in about a 35% reduction in the strength at age 28 days.

One interesting point, not all tested mixtures exhibited compressive strength lower than 2.1 MPa at age 28 days (i.e. upper strength limit in case future re-excavation is anticipated according to ACI 229 R). Only mixtures incorporating more than 10% agro-waste (i.e. M20, M30, and M40) did not exceed the 2.1 MPa at the age of 28 days. This can be ascribed to the low stiffness of the agro-waste compared to sand, along with the high voids induced by the high water content.

The removable modulus value was used to assess the excavatability, as shown in **Figure 5.6**. Typically, mixtures with a removable module less than one are considered easy to excavate manually [14]. The removable modulus is calculated based on the 28 days compressive strength (C) in (kPa) and its dry density (W) in (kg/m^3) using **Eq. 1**. As shown in **Figure 5.6**, although the increase in agro-waste has a significant effect on the achieved strength, yet all samples have a removable modulus (RE) above 1. Hence, the excavability for these mixtures manually is not easy. Mechanical equipment (i.e. backhoes) can be used to remove AAM-CLSM with a strength of less than 2.1 MPa [14]. Moreover, this excavatability limit is an arbitrary guideline that will depend mainly on the CLSM mixture constituents. Hence, this point will need further research to identify accurate limits. In conclusion, AAM-CLSM mixtures had the potential to accommodate higher amounts of agro-waste materials (i.e. even higher than 40%), which may result in manual excavable mixtures.

$$RE = \frac{W^{1.5} \times 0.619 \times C^{0.5}}{10^6} \quad \text{Eq.1}$$

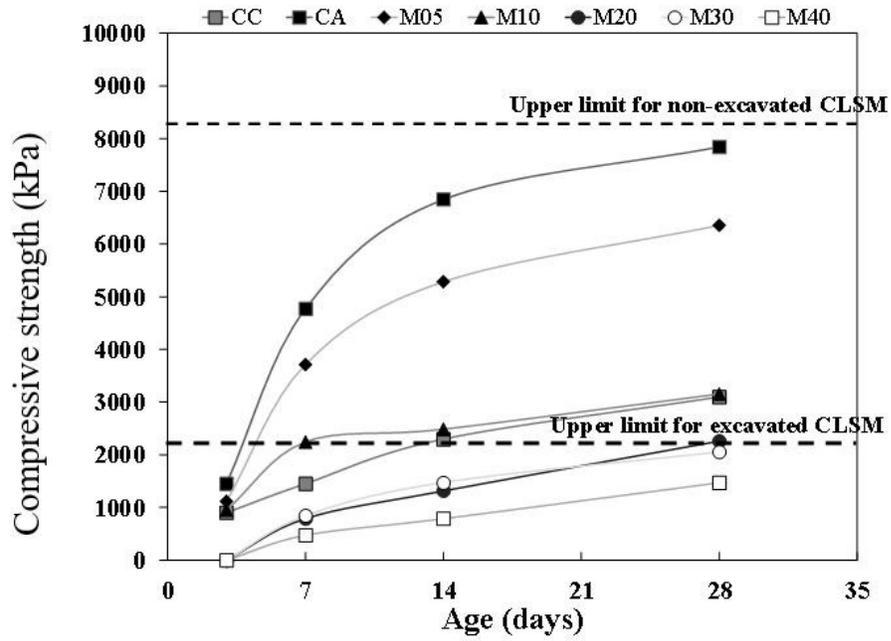


Figure 5.5 Compressive strength development for CLSM mixtures

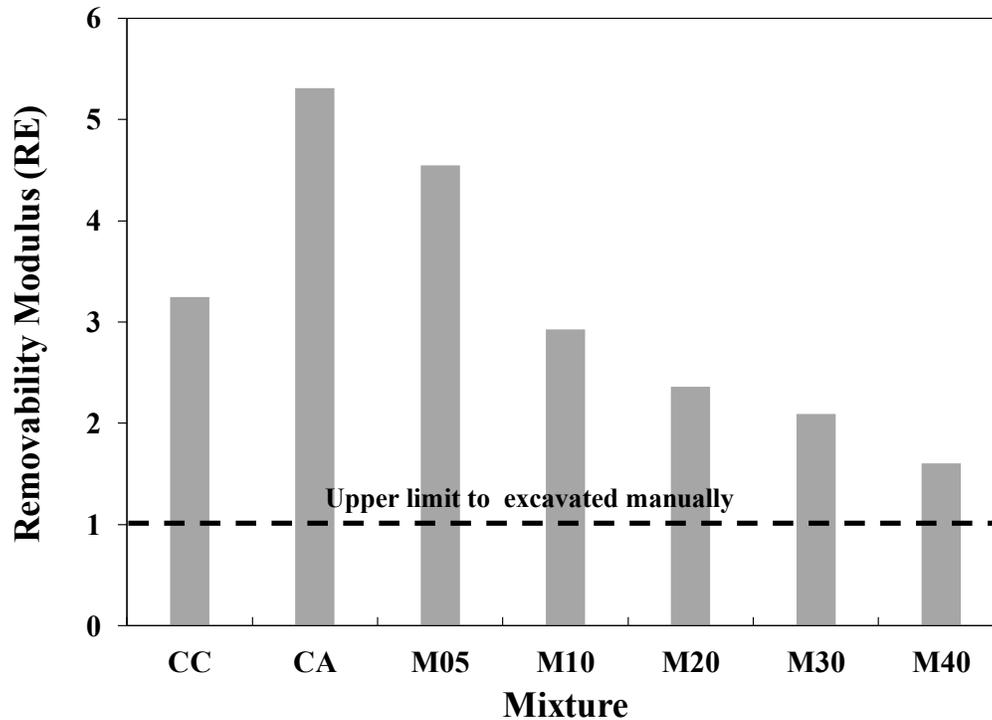


Figure 5.6 RE values for all tested CLSM mixtures.

5.3.4 Splitting Tensile Strength

Figure 5.7 presents the splitting tensile strengths for CLSM mixtures. As expected, AAM-CLSM exhibited higher strength than that of OPC-CLSM. In addition, tensile strength followed a similar trend to that for the compressive strength. Increasing agro-waste content in AAM-CLSM mixtures resulted in a lower tensile strength. For instance, AAM-CLSM without agro-waste exhibited a tensile strength of about 1635 kPa, while others with various agro-waste contents had tensile strength ranged from 235 up to 1070 kPa. **Figure 5.8** illustrates a good correlation between achieved tensile strength and corresponding compressive strength at age 28 days. This has also been observed in previous studies [131, 196]. The high amount of voids due to increasing water content or the porous structure of the agro-waste will result in a strain localization around the voids. This will lead to the carking formation in the interfacial transitional zone in the mortar around the void. Hence, the existence of the void will dominate the fracture behaviour for tested mixtures to a large extent [216].

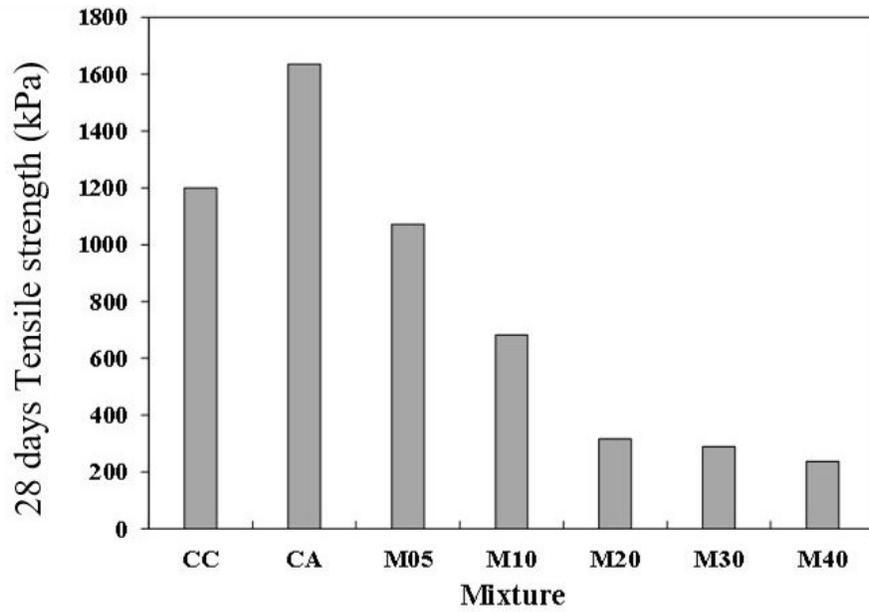


Figure 5.7 Tensile strength for CLSM mixtures at age 28 days.

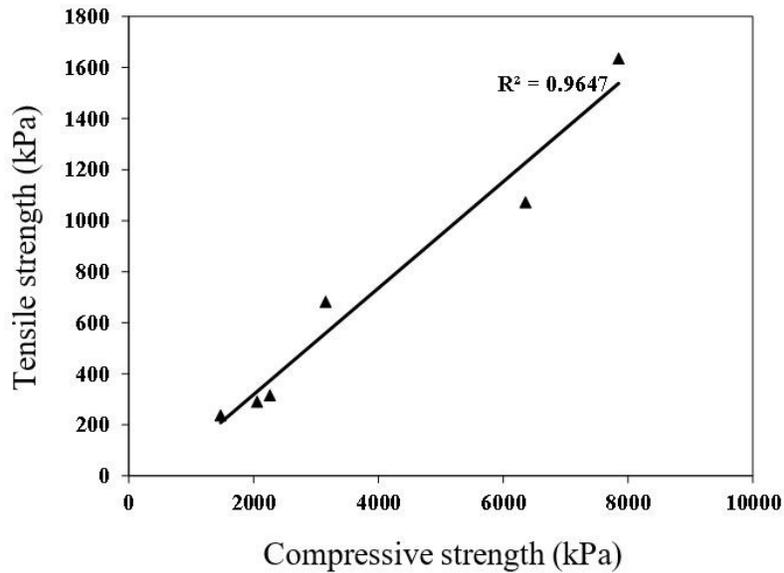


Figure 5.8 Correlation between splitting tensile strength and corresponding compressive strength.

5.3.5 Absorption Rate

Figure 5.9 illustrated the results of the absorption rate test for CLSM with and without agro-waste age of 28 days. AAM-CLSM exhibited a lower absorption rate than that of the OPC-CLSM; however, it showed a higher total porosity. This can be ascribed to the difference in the pore size distribution and functionality of each size range. The water absorption rate is directly related to capillary absorption, which reflects the water movement through small pores in the absence of any external applied hydraulic head. It is a result of the interaction between water and the pore walls [217]. According to previous studies, the pore diameters range for AAM porosity is out of the capillary pore range (i.e. 10 to 100 nm), which the main responsible for water transportation. Conversely, most OPC system pore size is within this range, leading to a higher rate of water absorption. This is in agreement with previous studies indicating that adding slag results in a notable reduction in the pore size and consequently the water transportation [218].

On the other hand, adding agro-waste was found to increase the total porosity and also the absorption rate. For instance, the absorption rate increased from 6.68% to 12.87%, as agro-waste content increased from 0% up to 30%. The higher the agro-waste content, the higher the measured absorption rate. One of the main features for agro-wastes is the porous (**Figure 5.10**), which results in a high-water absorption rate compared with sand. Also, the high initial water content will lead to a higher amount of voids at the hardened stage. This resulted in an open microstructure with high porosity[219]. Moreover, the low volume to the density ratio of agro-waste compared to that of the sand resulted in a lower volume density for CLSM [141]. Hence, the amount of internal pores that need to be filled by hydration products will increase. This will reduce the filling efficiency for the hydration process leading to porous CLSM mixtures. For instance, the porosity of the CLSM specimen with 20% agro-waste was about 7.83% higher than mixtures incorporating 10% agro-waste.

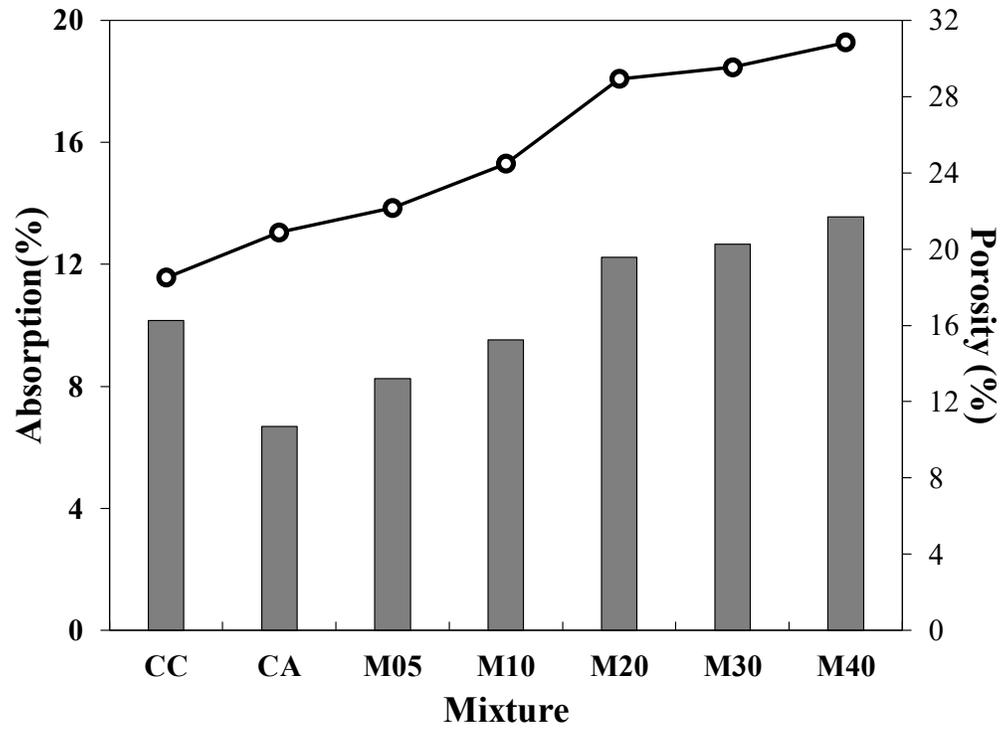


Figure 5.9 Absorption rate and total porosity for tested CLSM mixtures

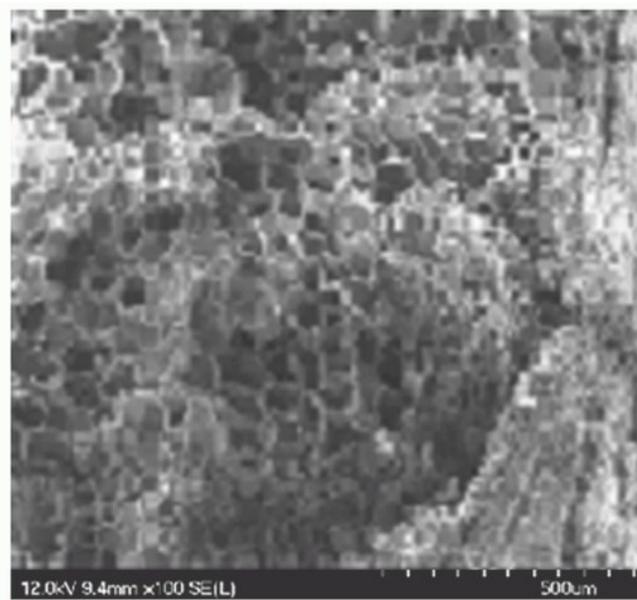


Figure 5.10 SEM showing the porous microstructure for agro-waste materials

5.3.6 Ultrasonic pulse velocity

Ultrasonic plus velocity test is a Non-destructive test that evaluates the homogeneity, the number of voids, and internal defects/cracks of concrete by measuring the speed of a wave through the material [220-222]. The ultrasonic pulse wave velocities (UPV) for all tested CLSM mixtures are shown in **Figure 5.11**. AAM-CLSM exhibited higher UPV than that of the OPC-CLSM, which was matching with previous compressive strength results. This can be attributed to the dual hydration process, as explained earlier. Generally, the higher the agro-waste content, the lower the measured UPV. For instance, increasing the agro-waste content from 5% to 20% results in around 18.5% reduction in the measured UPV. This can be attributed to the high porosity of the agro-waste itself, which will decrease the UPV [198]. One interesting point, the variations in measured UPV for mixtures with low water-solid ratio (w/s) were more sensitive than those mixtures with high ratios. For instance, for mixtures, M05 and M10, the w/s were around 0.94 and the reduction in the measured UPV, due to increasing the agro-waste from 5% to 10%, was about 11.45%. However, for mixtures M20 and M30, the w/s ratio was around 1.06, and the reduction in the measured UPV due to increasing the agro-waste from 20% to 30% was only around about 3.32%. This can be ascribed to the initial high-water content, which creates more void spaces in the cementitious matrix. Therefore, it seems that for high w/s mixtures, the amount of voids reached a threshold above which the reduction in UPV will be insignificant. Moreover, the changes in UPV were directly related to the achieved compressive strength [197]. This is confirmed by the good correlation between the 28 days compressive strength and UPV values ($R^2 = 0.975$), as shown in **Figure 5.12**. Accordingly, the higher UPV will reflect denser, compacted microstructure, and consequently better compressive strength.

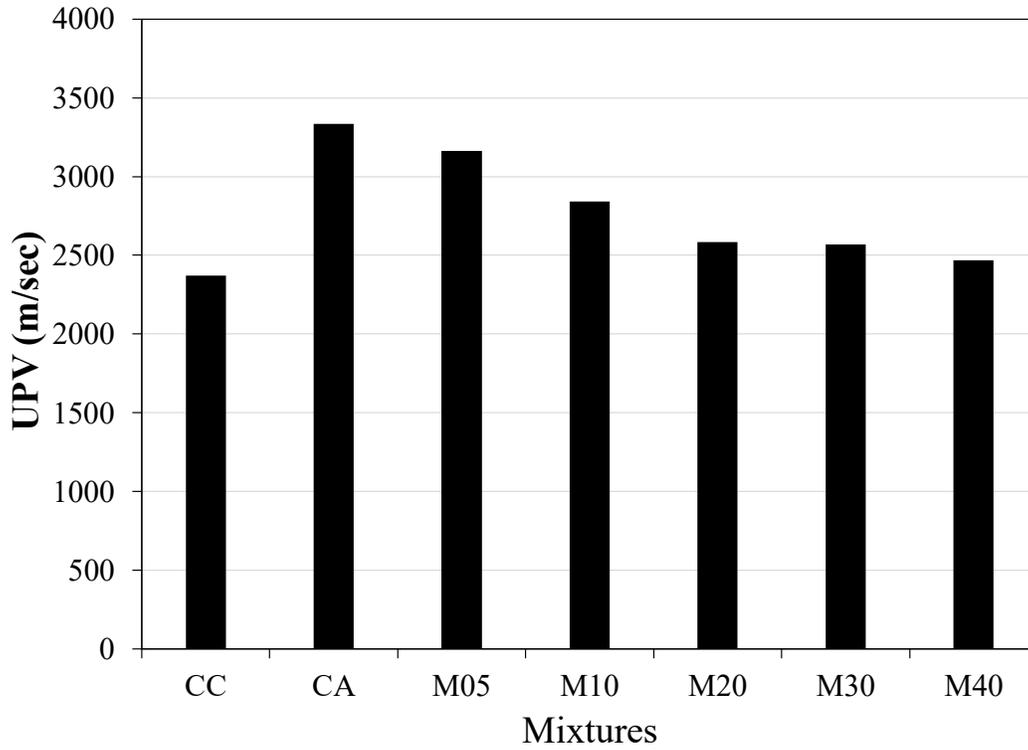


Figure 5.11 UPV for AAM-CLSM mixtures.

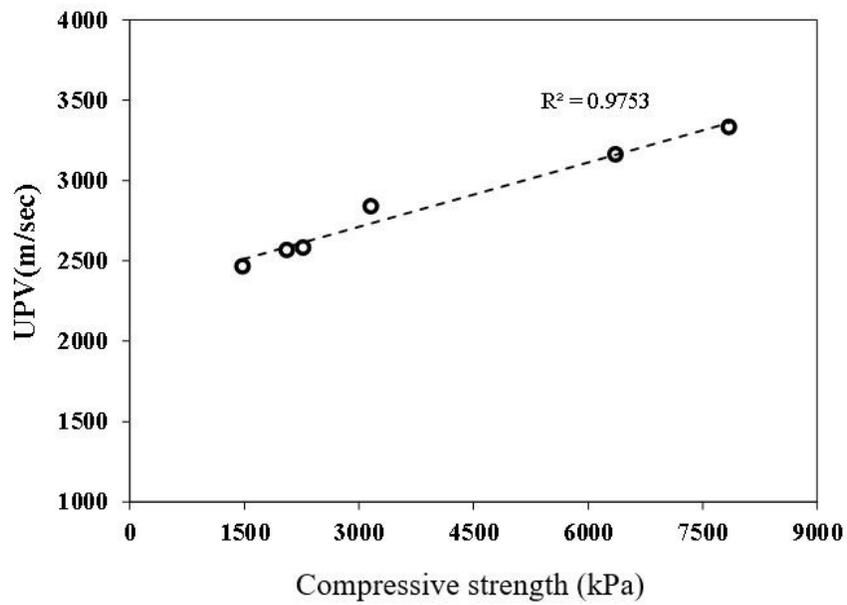


Figure 5.12 Relationship between measured UPV and 28 days compressive strength for AAM-CLSM mixtures

5.3.7 Sulphate attack

The performance of CLSM mixtures with and without agro-waste under sulphate exposure were evaluated based on the number of cycles in sulphate solution and residual strength. **Figure 5.13** shows the number of cycles sustained by CLSM specimens soaked in the sodium sulphate solution. AAM-CLSM did not show a better performance than that of the OPC-CLSM. Only two cycles were the difference between the two types of CLSM; however, the initial strength was almost double. This can be attributed to two compensating effects: 1) use of NaOH activator and 2) exposure to sodium sulphate solutions. AAM activated by NaOH was reported to have more degradation than AAM activated by other activators (i.e. water-glass) due to the presence of expansive phases such as ettringite and gypsum [223]. Conversely, submerging specimens in sodium sulphate is anticipated to increase mechanical resistance as it acts as an activation agent enhancing the geopolymerization process [224].

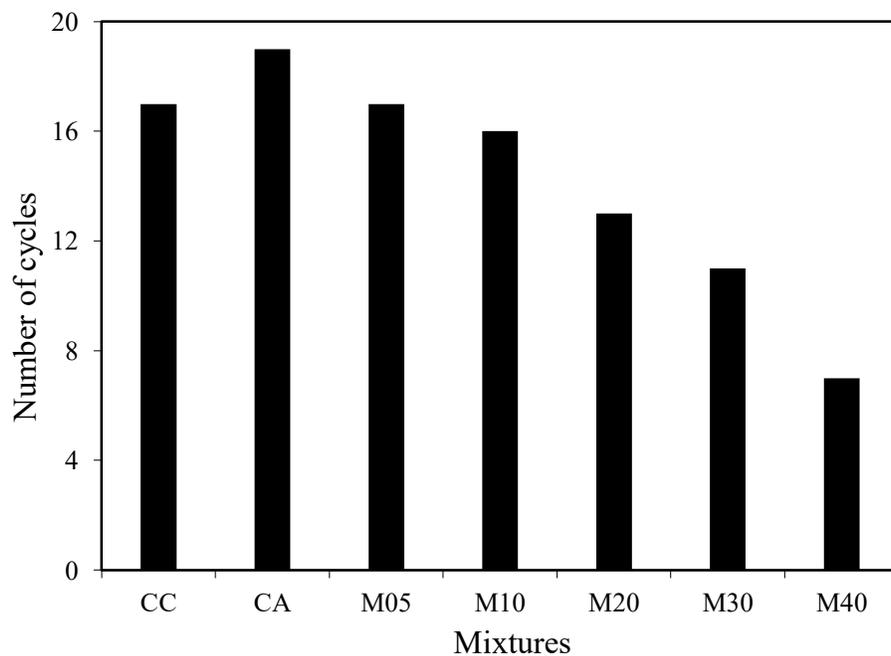


Figure 5.13 Numbers of sustained cycles in sulphate solution for all CLSM mixtures.

On the other hand, the higher the agro-waste content, the lower the number of sustained cycles. For instance, mixtures with agro-waste contents 5% and 20% sustained 16 and 11 cycles before failure, respectively. Reduction in strength of various CLSM specimens along 12 drying/wetting cycles in the sodium sulphate solutions is illustrated in **Figure 5.14**. Generally, the higher the

number of exposure cycles, the higher the strength loss. For instance, CA lost around 5% and 51% of its 28-days compressive strength after two and eight cycles, respectively. Moreover, adding agro-waste had a tremendous effect on the strength loss rate. For instance, M05 and M20 exhibited about 58% and 82% reductions in strength after eight cycles, respectively. Adding agro-waste had reduced the sulphate resistance for tested AAM-CLSM. This can be attributed to the increase in CLSM porosity, which facilitates the ingress of sulphate salt solution (i.e. an aqueous acid). This highly acidic environment will have two effects: 1) react with agro-waste and hydrolysis it [141], which was confirmed by the change in the submerging solution color; 2) affecting the stability of hydration products leading to de-polymerization of the alumino-silicate polymers and formation of zeolites[225]

It should also be mentioned that the cyclic wet and dry in the sodium sulphate solution is another parameter that is anticipated to accelerate the degradation of CLSM mixtures. Repetitive wet and dry cycles will induce a kind of volume instability (i.e. expansion during the wet period and shrinkage during the dry period). Consequently, internal stresses will develop, resulting in micro-cracking as it exceeds the CLSM tensile strength. Hence, these cracks will further propagate as alternate dry and wet conditions, which in turn break the internal structure ending up to specimen failure [202]. It is also expected that these micro-cracks if reach the surface, will facilitate more acidic to enter the CLSM and scale the effect of the pre-mentioned factors.

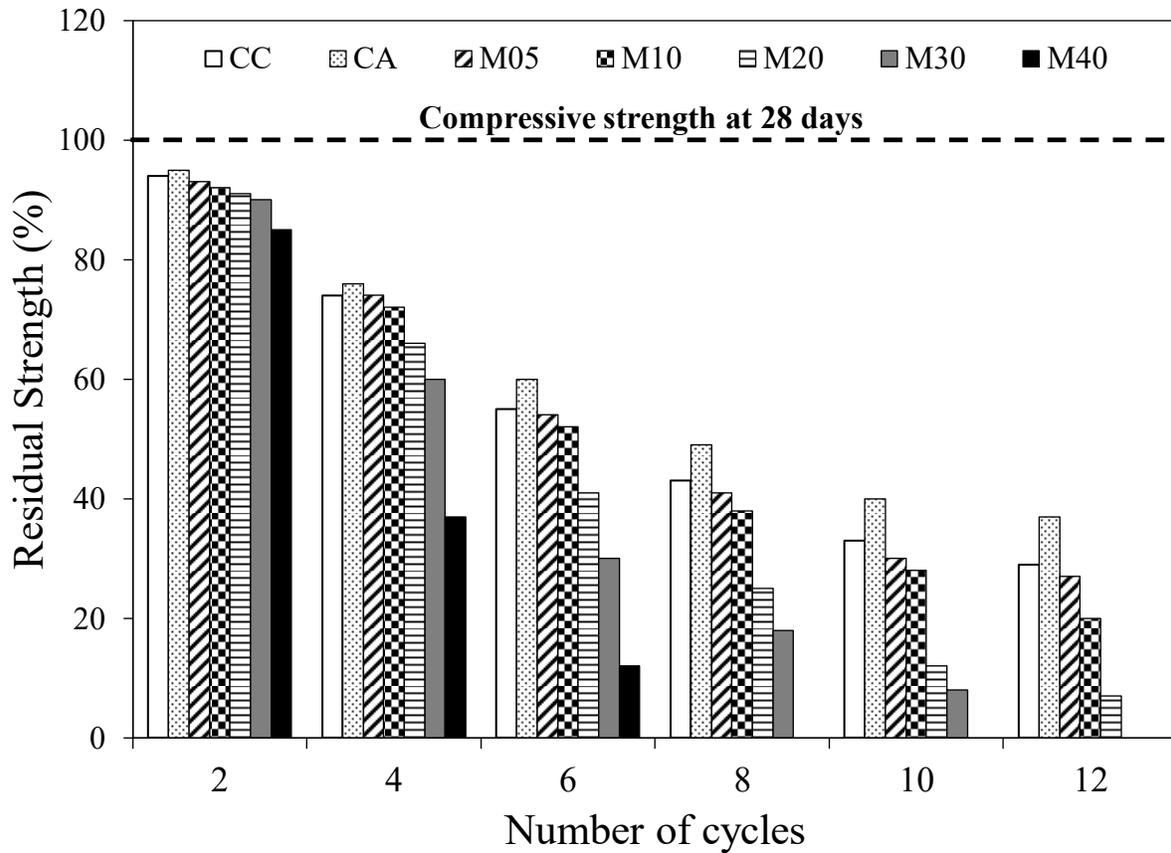


Figure 5.14 Residual strength for CLSM mixtures incorporating different agro-waste content.

5.4. CONCLUSION

This study focused on characterizing fresh and hardened of AAM-CLSM incorporating different percentages of agro-waste. The following conclusions can be drawn from results:

1. The high fly ash content in AAM-CLSM enhances flowability leading to lower water demand to produce CLSM with normal flowability. However, adding agro-waste will increase water demand due to its high water absorption and the increase in surface area.
2. Adding a high volume of agro-waste can be utilized to produce low-density CLSM for applications with concerns about the dead load.
3. AAM-CLSM achieve higher strength than that of OPC-CLSM due to the higher formation for hydration products as a result of geopolymerization and pozzolanic reactions. Adding agro-waste will reduce the strength, which aligns with CLSM strength requirements.

4. AAM-CLSM is hard to be manual excavate. The high value of agro-waste content (>40%) can be used to reduce strength leading to removability modulus less than 1.
5. Agro-waste had a porous microstructure, which will alternate the pore size range in AAM-CLSM. System. The higher the agro-waste content, the higher the porosity leading to a higher water absorption rate.
6. Activator for AAM-CLSM plays a vital role in controlling strength development and sulphate attack resistance for CLSM.

CHAPTER 6. CONCLUSIONS AND FUTURE WORK

By studying previous studies and examining agricultural waste samples, our research and experiments were divided into five sections. These studies have yielded significant results. These results could be a step towards future studies and to the eco-friendly construction industry. Here are the results:

1. Usage of agro-waste in the construction industry is an eco-friendly environmental solution that can be used in various industrial applications according to their physical and chemical properties.
2. One type of agro-waste in different forms can change the properties and behaviour of concrete.
3. Agro-waste can be used as a good alternative in most construction applications
4. AW1 and AW3, which were from the same group, changed concrete behaviour and properties regarding the appearance properties such as size.
5. Although the AWM2 appeared better in the first place than the rest of the wastes, it failed in the trial mixtures due to its significant water absorption capacity and was removed from subsequent observation.
6. Combining AWM1 and AM3 separately with agro-waste 4(fibre type) showed better results than samples with only one type of AWM1 or AWM3.
7. Fibrous Agro-waste can play different roles, such as reinforcement.
8. The study confirms the potential to produce CLSMs with high agro-waste content.
9. The low strength requirements for CLSM allows accommodating for a high amount of agro-waste (i.e., > 20%).
10. The reduction in the strength due to agro-waste addition will facilitate the removability of CLSM.
11. Increasing agro-waste will reduce bleeding; however, the absorption capacity of agro-waste must be considered to maintain adequate workability.
12. Although CLSM regarding the low amount of cement and the nature of it is known as a green construction material, it should be considered that in a high amount, the amount of

cement used can have a significant effect on the environment. This fact emphasizes the need for Alkali activated material CLSM, which is cement less.

13. The absorption of AAM-CLSM is based on the amount of agro-waste regarding the porous microstructure of agro-waste.
14. Adding agro-waste, which needs more water than more ordinary CLSM, makes a balance with high fly ash content in AAM-CLSM, leading to lower water demand.
15. Alkali activated CLSM shows higher results than OPC samples. The addition of agro-waste helps in reaching the CLSM strength limit (under 8.2MPa). About 40% or more replacement of agro-waste helps to reduce the strength of the final product helps to reach manual excavate range
16. In the sulphate attack and strength development of AAM-CLSM, activators play a significant role.

In this research, we have tried to examine all the aspects of using agricultural waste from the previous study to the effect on the behaviour and properties of the final agro-waste concrete after and, finally, their durability in corrosive environments. Among the things that can be done in future studies to complete this research are some suggestions that are presented in the following:

- 1) The low-strength concretes made in the third chapter of this study have shown good results in samples with fibrous agro-waste. Removing cement from the manufacturing process and reviewing agro-waste concrete could be among future considerations.
- 2) In addition to the durability in the corrosive area, the fire resistance, freeze/thaw cycles can be examined in the study of the durability of agro-waste samples.
- 3) One of the most popular reasons for using this waste in the construction industry today is the use of thermal insulation. Examining the thermal transfer of these concrete samples in different conditions (such as wet and dry) can attract much attention.
- 4) CLSM and AAM-CLSM sample's shrinkage can be one of the challenges that can be considered in the conditions of proximity to the water pipe during winter.

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