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The Effect of Agricultural Crop Residues and Bacteria on the Chemical and Engineering Properties of Eco-Cement Produced

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Abstract

Environmental concerns and issues are raised regarding Portland cement manufacturing. Besides, mortar and concrete deterioration is a very common problem. Therefore, eco-cement is an efficient alternative to Portland cement. Eco-cement is an environmentally friendly green building-material having self-healing abilities to remediate concrete cracks. In this study, eco-cement was produced from the ash of agricultural crops residues (pyrolysis of rice straw, sawdust of forest residues, and corn cob) which are hypothesized to enhance the binding abilities among the different components of cementitious materials (residues ash, cement, sand, and bacteria) and accelerate the biomineralization process to precipitate calcite and seal the concrete cracks. Through this study, 12 experiments (4 treatments in triplicates) were conducted. The resultant eco-cement was used to prepare mortar cubes which were tested and analyzed. The following engineering properties of the resultant eco-cement mortar cubes were investigated: compressive strength, four point bending test, and water absorption test. The results show that the addition of rice straw ash delivered the highest compressive strength and the highest four-point-bending of eco-cement mortar cubes average of 17.62 N/mm^2 and 6.13 N/mm^2, respectively; and an acceptable water absorption (5.82%) compared to all other treatments. The energy dispersive X-ray (EDX) analysis test results of the rice straw ash, sawdust ash and corn cob ash according to mass% of Si element were 7.55, 0.93 and 3.13 respectively and 0.84%, 0.45% and 0.41% for Al, respectively. It was concluded that the addition of the ash of agricultural crop residues to the cementitious mixture enhances its properties, where the resulting eco-cement is a promising substitute of the conventional Portland cement.

Keywords: Eco-cement; Building Materials; Microbially Induced Calcite Precipitation; Crop Residues; Green Buildings.

Introduction

One of the main sectors for development is the construction industry, which uses a lot of resources [1,2]. According to the International Energy Agency (IEA), the building and construction sector was in charge of 37% of CO_2 emissions and 36% of global energy consumption in 2020 [3,4]. Portland cement (PC) is a necessary component of concrete and mortar [5]. The worldwide cement consumption is anticipated to rise, from an estimated 4.1 Gt/a in 2019 to 6-13.5 Gt/a in 2050 [6,7]. Given the growing urbanization trend and rising global population, the need for cement for infrastructure development will rise in the upcoming years [5]. The significant carbon dioxide (CO_2) emission is the primary environmental impact of this increased cement use and concrete manufacturing. Cement manufacture involves chemical and thermal combustion processes that produce a significant amount of CO_2 and it is 8% of

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the world's CO_2 emissions [8,9]. Apart from the CO_2 emission, the cement factories also release numerous additional greenhouse gases, such as nitrogen oxides, sulphur oxides, and others [9]. Therefore, Cement replacement is urgently needed to reduce greenhouse emissions (GHGs) like CO_2 [10,11,12]. Hence, In order to address these resource conservation-related issues in the construction industry and bring the production of concrete and mortar within the purview of sustainable production processes, it is necessary to investigate various techniques [2].

In 2008, Egypt has issued its National Sustainable Development Strategy (NSDS). The strategy addressed many priority areas and challenges related to economic, social, institutional, and environmental aspects of the society. The strategy's main areas of focus were urban growth, solid waste management, and industrial development [13]. On the three pillars of sustainable development, Egypt's Vision 2030 is based: the economic dimension, the social dimension, and the environmental dimension. By maintaining the environment, enhancing infrastructure, and relying more on renewable energy for sustainable production, this study embodies Egypt's 2030 vision. Recent interest focused on sustainable development and the recognition of eco-concrete with the population growth around the world. Researchers are becoming increasingly concerned about concept of a green economy, which is important to the environment and society [14]. Since green building materials are created from eco-friendly resources and utilized to create eco-constructions that are subject to eco-design, the green building has an environmental component. This allows taking down the building components and materials, after the building lifetime, to environmentally friendly materials that can be re-used or recycled. The "Green Building" includes several sub-topics such as: green building materials (e.g., eco cement), green designs, green roofs, and green technologies [15]. However, in conventional buildings, Portland cement is commonly used as building material. Nevertheless, due to several environmental concerns and issues raised when Portland cement is produced and used, eco cement is alternatively used in green buildings as novel green building-material and energy-saving material [16].

To pave the way for sustainable development, Cement has been replaced with industrial byproducts like fly ash and slag. Not only can it reduce carbon dioxide emissions, but it can also effectively utilize these byproducts. Along with these industrial wastes, renewable and sustainable resources such agricultural waste ashes are attracting interest due to their usefulness as additional cementitious ingredients [17,18]. This is primarily because these leftover ashes in the concrete mix are actually readily available, inexpensive, and highly reactive [18,19]. Furthermore, the agricultural waste is typically disposed of in methods that are unfriendly to the environment. For example, Landfilling results in occupying valuable land and disrupting the ecosystem, while open burning degrades the environment and contaminates groundwater resources [5,20,21]. In recent years, some bio-waste have been extensively incorporated as alternative cementitious materials in concrete such as rice husk ash, rice straw ash, sugarcane straw ash, sugarcane bagasse ash, corncob ash, wood ash, wheat straw ash, coconut shell ash, palm oil fuel ash, groundnut husk ash, cassava peel ash, elephant grass ash, tobacco ash and bamboo leaf ash [1]. Most of the agricultural wastes contain high content of silica, when burned at a controlled temperature and environment, this crystalline silica transforms into reactive amorphous silica, which can contain up to 90% silica and has strong pozzolanic properties [5,18,22]. The creation of extra calcium silicate hydrate (C-S-H) as a result of the pozzolanic reaction of the treated agricultural residues with cement hydrates and $Ca(OH)_2$, increases the strength and durability of the resulting cementitious mixture [5,23]. In general, using biomass ashes in concrete and cement promotes sustainable development and zero-waste technologies [18].

On the other hand, Concrete and stone used in construction are exposed to the weathering effects of numerous physical, chemical, and biological variables [24]. All building materials are porous, and this porosity, together with moisture and other dangerous compounds such as chlorides, sulfates and acids affect the material and decrease their strength and life [25]. These issues are basic motivations to introduce new strategies for protecting and consolidating various building materials and healing concrete cracks [26]. Recently, an environmentally friendly strategy has been employed to manufacture bio-concrete by the usage of microorganisms in the traditional concrete to enhance its durability and compressive strength [23]. A bio-geochemical mechanism called microbiologically generated calcium carbonate precipitation (MICP). In this technique, $CaCO_3$ in the presence of Urea and $CaCl_2$ is produced. The produced $CaCO_3$ helps to improve the different properties of cement like compressive strength and water absorption, has potential to block the pores in concrete [26]. The main categories of microorganisms that can result in carbonate precipitation are photosynthetic microorganisms including Cyanobacteria and Microalgae, Sulfate-reducing bacteria and a few species of microorganisms engaged in nitrogen cycle [27]. Therefore, Future uses of bio-induction and bio-remediation techniques in cementitious composites appear potential for the development of sustainable infrastructure [28,29].

In this study, eco cement was produced from the ash of agricultural crops residues (pyrolysis of rice straw, sawdust, and corn cob) which are hypothesized to enhance the binding abilities among the different components of cementitious materials (residues ash, cement, sand, and bacteria) and accelerate the biomineralization process to precipitate calcite and seal the concrete cracks.

The main objective of the present study is to use agricultural crop residues and bacteria for producing eco cement as an environmentally friendly green building material and an efficient alternative to Portland cement. This general objective can be further elaborated in terms of the following specific objectives:

1. Using the ash of agricultural crop residues and bacteria in the Portland cement mixture to produce eco cement, while maintaining the properties of the produced eco cement.
2. Investigating the following engineering properties of the resultant eco cement: Compressive Strength, Four-Point-Bending Test, and Water Absorption Test.

2. Material and methods

2.1. Experimental Setup

2.1.1. Isolation of calcium carbonate ($CaCO_3$) precipitating bacteria

Two samples of soil and sludge were used for isolation of bacteria. The soil sample used was a clay soil obtained at depth 30 cm under the corn plant from Plant Production Department, Faculty of Agriculture, Cairo University; and the sludge sample was obtained from Agricultural Experiment and Research Station. These samples were used for isolating calcium carbonate ($CaCO_3$) precipitating bacteria using the dilution plate technique. Ten grams of soil and sludge samples were suspended in 90 ml of a sterile saline solution (0.85% NaCl) separately and shaken thoroughly for 10 min. Serial dilutions of each sample were prepared using sterilized saline solution. Suitable dilutions were plated on the solid medium of precipitation agar medium (containing nutrient broth (3 g/l) supplemented with urea (20 g/l), $NaHCO_3$ (2.12 g/l), NH_4Cl (10 g/l), $CaCl_2.2H_2O$ (28.5 g/l), agar (15.0 g/l) and adjusted pH to 7) for soil sample and nutrient agar and adjusted pH to 8) for sludge sample, then the poured plates were incubated at 28°C for between 24 and 48 h, respectively. The pure bacterial colonies were grown on nutrient agar slant, kept in a refrigerator at 5 °C, and sub-cultured monthly for continued study by [30,31,32,33].

The selected isolates were grown in nutrient broth-urea (NBU) medium (containing 8 g/L nutrient broth supplemented with 2% urea, 25 mM $CaCl_2$, and adjusted pH to 7) for 48 h at 37 °C under shaking condition (130 rpm) as described by [30,31,34]. The bacterial type was identified with molecular testing.

2.1.2. Agricultural Crop Residues

The following agricultural crop residues (rice straw, sawdust, and corncob) were used in this study as bio-silica sources and feedstocks for eco cement production. The agricultural crop residues were obtained from Faculty of Agriculture, Cairo University and Agricultural Experiment and Research Station. Crop residues were burned by the pyrolysis process. Pyrolysis is the process of thermochemically decomposing organic material at extremely high temperatures without oxygen, is applied to avoid negative environmental impacts of residues burning. The residues were placed separately in the muffle furnace at 700°C for 5 hrs. The incinerated products were then ground to obtain a homogeneous size. The produced ash was later added to the cement.

2.1.3. Preparation of Mortar Cubes

Mortar is composed of cement, sand, and further aggregates such as fly ash. The addition of water activates the cement, which is the element responsible for binding the mix together to make one solid object. In the following preparation process, agricultural crop residues containing bio-silica were pyrolyzed and used as a substitute of fly ash. Pyrolysis is applied to avoid the negative environmental impacts of residues burning.

This preparation process was conducted as described in the literature [1,30,32,35], but with some amendments. The preparation steps are mixing, casting, and curing. It starts with dry mixing of all the solid components (cement, sand, and crop residues ash) with a mortar blender. Water and bacterial culture were then added to the dry mixture and the mixture which was further mixed at 140 rpm until it becomes homogenous. The specification of Portland cement used is (CEM I grade 42.5 N) conforming to international standards was used and blended with sand and pyrolyzed agricultural crop residues. The cement-to-sand ratio is planned to be 1:3 (by weight), and the water-to-cement ratio is 0.5, and the ash-to-cement ratio is 0.10. Cement, sand, and residues ash were then meticulously blended together with water and grown culture of bacterial isolate corresponding to OD600 0.5. Cubes mold of dimension 70 mm × 70 mm × 70 mm were prepared for compressive strength test and water absorption test. Prism molds of dimension 40 mm × 40 mm × 160 mm were prepared for four-point bending test. Cubes and prisms were cast and compacted in a vibration machine. After de-molding, all specimens were cured in nutrient broth-urea (NBU) medium at room temperature until compression testing at 28 days except control samples were cured in water. Media were replenished at a regular period of 7 days. Control samples (standard Portland cement not including bacteria and ash of crop residues) were also made in an analogous method. Four treatments with two curing methods were implemented in this study as specified in Table 1.

Table 1

Specifications of the specimens

Specimen	Composition	Mechanism of Curing
Control	Cement: sand = 1:3 Water/cement = 0.5	Water Curing for 28 days
Eco cement **=** Control **+** Bacteria **+** waste ash(rice straw ash or sawdust ash or corn cob ash)	Cement: sand = 1:3 Waste ash/ cement = 0.1 Bacterial culture/cement = 0.5	*NBU medium with urea and $CaCl_2$ curing for 28 days

*NBU: nutrient broth-urea

2.2. Measurements and Analytical Methods

2.2.1. Compressive Strength

Compressive Strength is the resistance of a material to breaking under compression. This test was conducted as described in literature [31,32,36] in order to find out the compressive strength of the mortar cubes against vertical loading. Compression testing was conducted after 28 days of de-molding and hardening using automatic compression testing machine. The specimen was fed into the machine and progressively loaded until it cracked. The final load was then recorded. The values are expressed in (N/mm^2).

2.2.2. Four-Point-Bending Test

Eco cement mortars exhibit mechanical behavior which can be evaluated for tensile strength, stiffness, crack strength, peak stress, and work of fracture as a measure for strain during multiple cracking where these can be investigated using a four-point-bending test. After 28 days the specimens were cracked using the standardized four-point-bending test [35]. The cracking took place under standard laboratory conditions at a relative humidity of 60%. A servo-hydraulic testing machine ensured a displacement-controlled test (0.002 mm s^{-1} to imitate a quasi-static load). The vertical displacement was distorted with this loading speed. The values of four-point-bending are expressed in (N/mm^2). Four-Point-Bending of the specimens was determined by using following formula:

$$\text{Flexural Strength} = Pl/ bd^2 \quad (1)$$

where: Flexural Strength in Pa; P = load in N; l = span length between supports in m; b = width of prism at point of fracture in m; and h = height of prism at point of fracture in m.

2.2.3. Water Absorption Test

The water absorption test was conducted as per ASTM C642-97 [37,38,32] to find out the increase in resistance to water infiltration in mortar. The cube molds of 70 mm were made both with and without bacteria, crop residues ash. The mortar specimens were cured for 28 days. After curing, their saturated masses after immersion were calculated. Then the specimens were dried at 110°C in furnace, creating a mass balance of lower than 0.5% between 2 measurements at 24 h intervals. Water absorption of the specimens was determined by the following formula:

$$\text{Water Absorption } \% = [(W1-W2)/W2] \times 100 \quad (2)$$

Where: W1 is the mass of the sample after immersion and W2 is the mass of the oven dried sample.

2.2.4. Calcite Content

Calcite content was assessed using the gravimetric analysis of acidified samples. 10 g of the powder sample was implemented after oven-drying at 105 °C for 24 h. Later 2 M of HCl was poured onto the prepared powdered, where CO_2 released because of the reaction between calcite and HCl. The residue was gathered and oven-dried again, and the weight loss was determined before and after acid rinses as described by [39], the results were used to determine the percentage of calcite content in the specimen as described by [26,40].

$CaCO_3 + 2\ HCl \rightarrow CaCl_2 + H_2O + CO_2$

2.3. Statistical analysis

These data were analyzed based on standard division (SD±) using Microsoft Excel program version 2010.

3. Results

3.1. Bacterial Identification

In this study, the bacterial culture identification using DNA analyses was identified; they were Sporosarcinapasteurii and Bacillus sp., which were rod-shaped, spore-forming and Gram-positive.

3.2. Scanning Electron Microscopy (SEM)

The morphology of samples was examined by the scanning electron microscope (SEM) for rice straw ash, sawdust ash and corn cob ash are presented in Figure 1A- C.

A **B** **C**

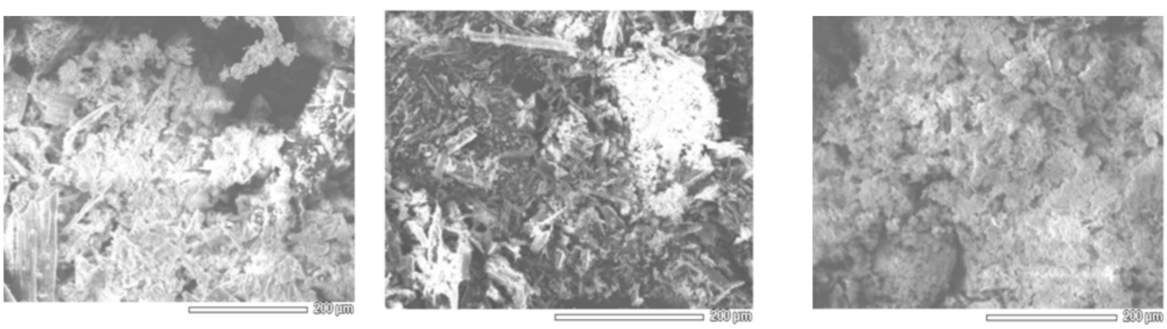

Fig. 1. SEM images for rice straw ash (A), sawdust ash (B) and corn cob ash (C) (Magnification: 240 x)

3.3. Energy Dispersive X-Ray (EDX) Analysis

EDX spectrum on a SEM (Figure 2, Figure 3A-C) examined the chemical structure of rice straw ash, sawdust ash and corn cob ash. The sample contains many components like K, Cl, Ca, O, C, Si and Al, the expected components from those materials were silica (SiO_2), alumina (Al_2O_3), ($CaCl_2$) and (K_2O).

Figure 2 shows that rice straw ash had the highest percentage of silicon (Si) with percentage (7.55), then corn cob ash with percentage (3.13) then sawdust ash with percentage (0.93) and for aluminium (Al) the highest percentage was existed in rice straw ash with percentage (0.84), then sawdust ash with percentage (0.45) then corn cob ash with percentage (0.41). Due to the importance of the presence of silica and alumina among the basic compounds in the cement composition, the cement sample containing rice straw ash was the best because it was the highest in terms of the elements silicon and ammonium. Thin film standardless quantitative analysis fitting coefficient for rice straw ash, sawdust ash and corn cob ash were 0.0350, 0.0766 and 0.0709, respectively.

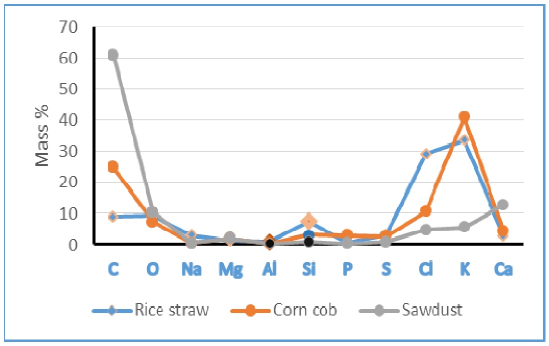

Figure 2 . EDX results of rice straw ash, sawdust ash and corn cob ash

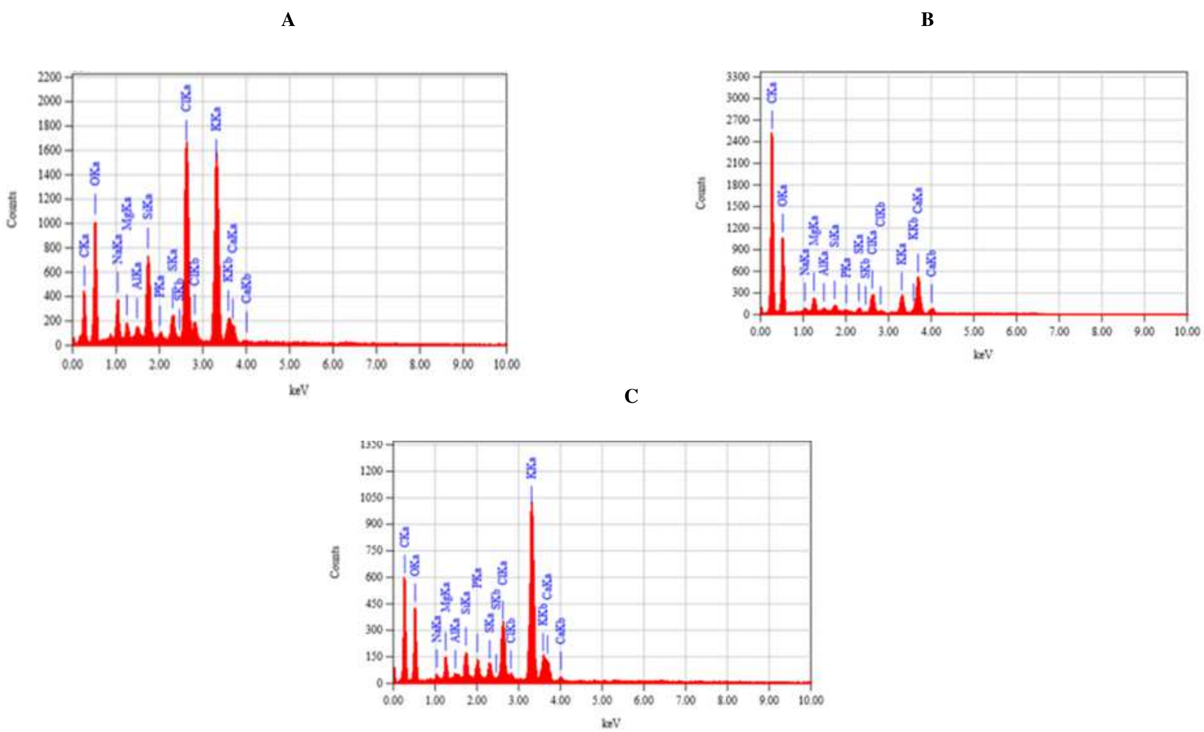

Figure 3. EDX results for rice straw ash (A), sawdust ash (B) and corn cob ash (C)

3.4. Effects of ash additives on compressive strength of eco cement mortar cubes

The effects of different ash additives of rice straw, sawdust, and corn cob on the compressive strength of eco cement mortar cubes were evaluated. Figure 4 shows the compressive strength of eco cement mortar cubes after ash addition compared with the control (Standard Portland Cement), where the addition of rice straw ash delivered the highest compressive strength of eco cement mortar cubes and, however, the control delivered the lowest compressive strength. Noting that, the higher the compressive strength, the better the eco cement mortar cube.

3.5. Effects of ash additives on Four-Point-Bending of eco cement mortar cubes

The effects of different ash additives of rice straw, sawdust, and corn cob on the four-point-bending of eco cement mortar cubes were evaluated. Figure 4 shows the four-point-bending of eco cement mortar cubes after ash addition compared with the control

(Standard Portland Cement), where the addition of rice straw ash delivered the highest four-point-bending of eco cement mortar cubes and, however, the addition of corn cob ash delivered the lowest four-point-bending. Noting that, the higher the four-point-bending, the better the eco cement mortar cube.

3.6. Effects of ash additives on water absorption of eco cement mortar cubes

The effects of different ash additives of rice straw, sawdust, and corn cob on the water absorption of eco cement mortar cubes were evaluated. Figure 4 shows the water absorption of eco cement mortar cubes after ash addition compared with the control (Standard Portland Cement), where the addition of corn cob ash delivered the lowest water absorption of eco cement mortar cubes and, however, the control delivered the highest water absorption. Noting that, the lower the water absorption, the better the eco cement mortar cube.

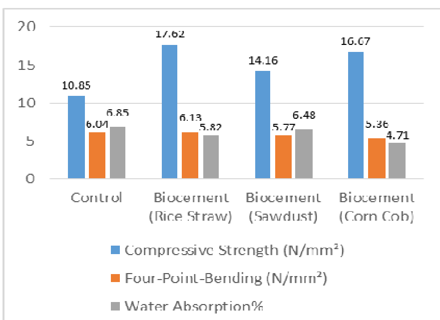

Figure 4. Results of Compressive Strength Test, Four -Point - Bending Test, and Water Absorption Test

3.7. Calcite Content

Upon hydrochloric acid solution addition, it reacted with calcium carbonate precipitate and, therefore; an eruption was observed, and carbon dioxide gas was released as shown in Figure 5.

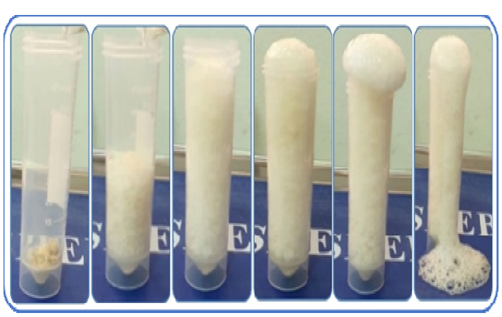

Figure 5. Eruption and CO_2 release after adding HCl to the precipitate which indicates the formation of $CaCO_3$

4. Discussion

This research represents one of the unique solutions of many problems facing the world these days. In this approach agricultural crop residues are being used as a substrate for eco cement production. The world produces millions of tons of agricultural wastes every year around the world which demands a radical solution to not only get rid of these wastes in an ecofriendly method but also to convert these wastes into a high-value product, this will be positively reflected on the economy. Based on the results of this study, the addition of the ash of agricultural crop residues to the cementitious mixture is an easy and affordable technique with a very good outcome. Eco cement production using agricultural wastes is an environmental-friendly method and will encourage to scale-up the process to an industrial scale to generate clean cementitious materials using an ecofriendly technique and solve the agricultural wastes issue as well.

Eco cement production using agricultural wastes is an environmental-friendly method and will encourage to scale-up the process to an industrial scale to generate clean cementitious materials using an ecofriendly technique and solve the agricultural wastes issue as well. Besides, eco cement is a valuable technique to mitigate the CO_2 released through cement production. Eco cement decreases clinker and energy consumption as well as CO_2 emissions, where the raw feedstocks for eco cement production are renewable bio mass. In this study, it was found that the final eco cement mortar has and improved performance, which agrees with the statements of [1,5,16]. Besides, it was found that eco cement mortar has better resistance to water permeability than cement from Portland cement alone, which agrees with the findings of [41].

In this study, eco cement was produced as a blend of bio-silica, found within the ash of crop residues, with Portland cement, where the ash production process was integrated into energy production to simultaneously gain energy and bio-silica ash since pyrolysis has been applied, which agrees with [16] except that they used combustion but not pyrolysis as the present study. The pozzolanic reaction of the agricultural waste ashes with the cement hydrates is assumed to be the cause of the increase in compressive strength of blended mixtures (Portlandite). Furthermore, the pore filling effect of the small particles of these ashes also contributes to the increase in compressive strength which agrees with the concept of [5].

On the other hand, in this study eco cement was produced as a mixture of bacterial culture and biomass which was produced from cheap raw materials (agricultural crop residues) with inorganic chemicals (sand, aggregate, and standard Portland cement), where this agrees with production process applied by [42]. The addition of bacteria and agricultural waste ashes improve the mechanical properties of concrete. This can be as a result of the reduction of pores in the microstructure of the concrete by filling micro-pores, decreasing the potential entrance of acids and chemicals into the concrete, which agrees with [30,43]. Furthermore, the ash of agricultural crop residues was used as replacement for limestone, sand, clay, and iron slag as the raw materials to produce eco cement, which agrees with the concept of [42]. An important key issue is that the bacteria used to produce the eco cement were found to precipitate calcite that seals cracks usually found in common concrete and mortar, which agrees with [44]. Urease helps in mineralization of $CaCO_3$, by hydrolyzing urea available in the environment. It releases carbon dioxide from urease activity, bacteria can use urea as sole nitrogen source and produce NH_3 which increases the pH in the environment causing Ca^{2+} and CO_3^{2-} to precipitate as $CaCO_3$ which seals concrete cracks. Urease producing microbes keep on surviving and growing within the concrete structure after the primary utilization, which agrees with [25]. Moreover, these bacteria which were added to the mixture of eco cement, improved its compressive and flexural strength as well as its ability to remediate cracks, where microbially induced carbonate precipitation (MICP) was implemented for the remediation of cracks, which agrees with the statements of [24]. As a result, bacteria were found to be self-healing agents for the autonomous reduction of permeability of concrete upon crack formation. Eco cement can be used for remediation of cracks in cementitious structures through MICP that has various applications in remediation and restoration of a range of building materials such as mortar and concrete which agrees with [30].

According to the results of this study, it is evident the importance of silica and alumina particles in improving the properties of cement, where it was found that the higher the percentage of silicon and aluminium in the sample, the better the properties of cement. Accordingly, the eco -cement produced from rice straw ash has given the best results. In contrast, samples produced from sawdust ash were the least which agrees with [16]. The presence of high alumina and silica content in the sample contribute to the production of calcium aluminium silicate hydrate (CASH), which leads to the strength improvement in all the cement based materials which agrees with the concept of [1].

5. Conclusions

The performance of mortar mixtures included of bacteria and produced with partial replacement of cement with agricultural waste ashes of rice straw, sawdust and corncob were evaluated in this investigation. The compressive strength test, four-point-bending test, water absorption test, scanning electron microscope (SEM) and energy dispersive X-Ray (EDX) were conducted to study the effect of addition the agricultural waste ashes and bacteria on properties of cement. Based on the experimental results, the conclusions can be made as follows:

- It can be concluded from the results of this study that the addition of the ash of agricultural crop residues and bacteria to the cementitious mixture improves its properties especially in terms of the compressive strength, the four-point-bending, and the water absorption. On the other hand, this process reduces the energy consumption as well as the emissions of greenhouse gases which causes the global warming and then the climate change.
- The results show that the addition of 10 % rice straw ash delivered the highest compressive strength and the highest four-point-bending of eco cement mortar cubes average of 17.62 N/mm^2 and 6.13 N/mm^2, respectively; and an acceptable water absorption (5.82%) compared to other control treatments.
- For the EDX results of the rice straw ash, based on the silica content and aluminum are 7.55 and 0.84 (mass%). For sawdust ash, Si is 0.93% and Al is 0.45%. On the other hand, Si and Al are 3.13% and 0.41% for corn cob ash.

For the effective use of agricultural biomass ashes in cement concrete, further focused study in this field is required. It appears to be a crucial and promising contribution towards the sustainability of the construction industry.

6. Conflicts of interest

There are no conflicts to declare.

7. Formatting of funding sources

There are no funding sources.

8. Acknowledgments

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