Experimental Investigation on the Effects of Coffee Husk Ash as Partial Replacement of Cement on Concrete Properties

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The production of cement results in the depletion of natural resources and consumption of huge energy and CO₂ emissions to the environment that cause global warming and climate change. To alleviate the problem, there is a growing interest of researchers to find an alternative material to partially cement by industrial and agricultural wastes. This research was therefore aimed to examine the potential of using coffee husk ash (CHA) as partial replacement for ordinary Portland cement (OPC) in C-25 concrete production. Concrete mixtures were prepared by partially replacing cement with CHA in different proportions (0%, 5%, 10%, and 20%) by weight with a constant w/c of 0.5. The consistency, setting time, workability, compressive strength, water absorption, sulphate attack, Fourier transform infrared (FTIR), and thermo gravimetric (TGA) tests were conducted on concrete samples. The test results revealed that the workability of mixtures showed a decreasing trend with an increase in the share of the CHA content in which the measured slump values ranged between 15 and 35 mm. Contrarily, the setting time of concrete mixtures showed an increasing trend with an increase in CHA content. The initial and final setting times were in the range of 67–126 minutes and 310–524 minutes, respectively, which is in the range of the standards. The compressive strength of concrete decreased with an increase in the share of the CHA, in which the results were measured in the range of 35.1–22.7 MPa for the 28th day sample with 5% and 20% of CHA, respectively. However, it increases as the curing days increase, whereas the water absorption of the concrete increases as the CHA increases but decreases as the curing days increase due to the porous nature of the CHA. From the study microstructure of concrete using Fourier transform infrared spectroscopy and thermogravimetric analysis, differential thermal analysis results show that the C-S-H gel would be from 950–1100 cm⁻¹ wavenumber and at 500°C, and the CHA sample was decomposed and mass loss was observed at 500°C. In general, it was also observed that, from the compressive strength, the concrete satisfies its design strength up to 10% replacement level without compromising the performance of concrete.

1. Introduction

Nowadays, construction has become the most crucial component in the development of a country, and it plays an important role in the social, economic, and especially in the creation of employment opportunities. When we talk about construction, it is directly or indirectly related to concrete production and which leads to massive extraction of natural resources [1, 2].

Concrete, as the primary and the world’s oldest and most widely used construction materials, is relatively inexpensive to produce due to the abundance of its components, which include aggregate and sand, as well as cement and water as the binder. Due to the general abundance of its raw materials, cement production is widespread. Even though concrete has a relatively high durability, good aesthetics, ability to withstand extreme weather conditions, and is easy to use, it will almost certainly remain the preferred construction material [3].

In concrete production, the paste is made of cement and water, as well as other cementitious and chemical admixtures, whereas the aggregate is made of sand, gravel, or
crushed stone. The paste binds the aggregates together. The aggregates are relatively filler materials that comprise 70% to 80% of the concrete by mass and can thus be expected to influence its properties [4].

The binder and most expensive and environmentally unfriendly element of concrete is cement, which is an ecologically unfriendly process due to the release of CO₂ gas into the atmosphere and ecological degradation. Portland cement clinker production is a major source of CO₂ and other greenhouse gases with the contribution of 5–7% of the annual global atmospheric CO₂ emission [5].

Therefore, there is a need to change cement production practices in order to reduce CO₂ emissions, extensive use of natural resources, as well as lowering overall construction costs through reduced cement consumption and utilization of wastes as partial replacement of cement. In addition to cement replacement, based on the testing and research carried out on wood wastes such as particleboards made from fiber and bagasse with adhesive, optimum results have been obtained, and they can be utilized as raw material [6, 7]. The use of environment-friendly materials is an integral part of the modern cement utilization of fly ash, blast furnace slag, recycled aggregate, plastic waste, and supplementary cementing material for the production of eco-friendly green concrete.

Recently, some countries tested coffee husk ash (CHA) for its pozzolanic properties, which were discovered to develop some of the properties of paste, mortar, and concrete such as compressive, tensile, and flexural strength and durability properties in confident substitution percentages [9].

Regarding material abundancy, the International Coffee Organization (ICO) [10] confirms that Ethiopia is the fifth country of coffee producer in the world (440,580 metric tons) for the year 2019/20 next to Brazil (3,492,600 metric tons), Vietnam (1,829,220 metric tons), Colombia (846,000 metric tons), and Indonesia (685,980 metric tons).

Green coffee is produced either by dry processing or by wet processing. Following harvesting, the coffee fruits are separated from the pulp either through dry or wet processing. The dry process is simple and low-cost. The whole cherries are sun-dried in the open air before being separated from the hull (dried pulp and parchment) to obtain the green beans [11].

Coffee husk is the main solid residue from coffee processing that has no current commercial uses, and their proper disposal poses a serious environmental problem [12]. Coffee pulp is the first byproduct obtained during wet or semidy processing, accounting for 29 percent of the total dry weight of the cherry. Coffee pulp is made up of two parts: the exocarp (outer skin) and the mesocarp (fleshy portion) [13]. Coffee silverskin and spent coffee grounds are the main coffee industry residues obtained during the beans burning up and the process to prepare “instant coffee,” respectively. Because it is released during roasting if the beans are not polished prior to shipment, and coffee silverskin, also known as chaff, is the first coffee industry residue produced in consuming countries [13].

The physical properties of CHA obtained by different scholars depend on its processing parameters of particle size and specific surface area, relative density/specific gravity, bulk density, strength activity index, and texture [14]. Specific gravity of CHA reported by authors is very less (maximum of 2.72) when compared to the specific gravity of Portland cement that has a density of 3.15 g/cm³ [1, 8, 15–19].

The chemical compositions of CHA depend on temperature and burning time, chloride and moisture content, loss on ignition, and silica content [14]. The major chemical compounds of CHA decreased as the burning temperature of coffee husk increased. It is caused by the ash disintegrating into its constituent chemical elements [8]. Relative to other SCMs, CHA is composed of a small quantity of SiO₂, Al₂O₃, and Fe₂O₃ and higher amount of CaO, with different other oxides from which alkali content of K₂O is substantial (45–65%). and the loss on ignition (LOI) value of CHA is significant (Up to 20%) because of the carried out incineration and processing [1, 20–22]. For CHA to use as pozzolan in cement and concrete, it must fulfill requirements for the chemical composition as consistent with ASTM C618-19. As per ASTM C618-19, any substances with 70% or higher pozzolanic index may be used as a supplementary cementitious material.
The reason behind using pozzolan is the improvement found on both the fresh and mechanical properties of concrete. Lowering of the heat of hydration and thermal shrinkage, increase in water tightness, reduction in the alkali-aggregate reaction, resistance to sulphate attack, better workability, and cost efficiency are some of the improvements achieved by using pozzolan blended with Portland cement [1, 6].

Generally, concrete’s workability of fresh mixed concrete varies with the size, shape, and surface texture of particles; water-to-cement ratio, characteristics of ingredients, use of chemicals and minerals, and mix proportion. Upon the results of investigators, the workability of CHA measured both from the slump and compacting factor tests reduced with an increase in the share of the CHA [1, 8, 23]. Because of the absorptive nature of cellular CHA particles and the high fineness, concrete containing CHA requires more water for a given consistency (this increases its specific surface area) [1].

In addition, from the fresh mixed concrete tests of the consistency and the setting times results that with an increase in CHA amount, the consistency and setting time increase and are within the limit of ASTM standard [1, 8, 14]. The water absorption test results of a few authors revealed that a reduction in water absorption was observed for more CHA blended cement mortar, but it was higher than the control paste [23–25].

Regarding the compressive, tensile, and flexural strength of the concrete containing CHA, the strength increases with the increasing CHA content up to the optimum level [1, 8, 22–24, 26]. The reason for the decrease in concrete strength of cement partially replaced by CHA is because of the amount of CHA used above the optimum level, reduced cement hydration reactions because of CHA, curing times, and treatment solution [8, 23, 24].

Considering the above results of literature of few authors, partial replacement of natural resources by byproducts and wastes has also been discovered in consideration of the nonavailability of extracted natural resources to future generations and reducing the quantity and type of waste materials.

Previous research tried to investigate the effect of CHA on engineering properties of concrete. However, the effect of CHA on durability and microstructure of concrete is not yet sufficiently explored. Therefore, the main objective of the study under consideration is to determine the effects of engineering properties (consistency, setting time, slump for workability, compressive strength, sulphate resistance, water absorption and microstructure properties, and compressive strength relationships with that of sulphate resistance and water absorption) of concrete containing coffee husk ash as partial replacement of cement on the concrete properties.

2. Materials and Experimental Methods

2.1. Materials

2.1.1. Cement. The cement used is Dangote Cement Factory’s Ordinary Portland Cement (OPC) with a grade of 42.5R. The grading and physical properties are in conformity with the requirements necessitated by standard specifications of ASTM C 150 including consistency, setting times, and workability. The same type of cement was used for all the trial mixes, and the amount added per mix was kept constant.

2.1.2. Coffee Husk Ash. For this study, the coffee husk is collected from Zegie, Gojam, Ethiopia. Research was conducted on the burning of coffee husk and identified the suitable burning and residence time to be 500–600°C. The higher temperatures will give a higher amount of silica content, but the resulting silica is in the crystalline form that is not in an active state, and it is due to the disintegration of the ash to the respective chemical elements [8]. Therefore, the coffee husk sample of the study was exposed to the sun to eliminate surface moisture and was burnt in a carbonate furnace for three hours at 550°C to get the required CHA (Figure 1) then, to remove impurities and coarser particles; the ash particle size was reduced to the required level of fineness and sieved with a 75 mm sieve.

The chemical compositions of coffee husk ash (CHA) are mainly dependent on the ash processing parameters such as the burning method, duration and temperature of burning, separation process, and grinding. The chemical requirements of ASTCM 618 limit the sum of oxides SiO₂ + Al₂O₃ + Fe₂O₃ to 70%. The result of the chemical analysis of the CHA sample is given in Table 1. The chemical analysis indicates that the major oxide of CHA is SiO₂, Al₂O₃, Fe₂O₃, and CaO along with major loss on ignition (LOI). The loss on ignition (LOI) value for the CHA was found to be 32.88%, which was higher than that specified by the same standard. Moreover, the CHA was found to have high alkali content like K₂O (12.64%) implying the high potential for an alkali-silica reaction when used in concrete with silica reach aggregates, and hence, it would cause the reduction for the strength of the concrete by lowering the bonds between the paste and the inert materials as a whole [27]. As a result, rather than using CHA as a pozzolanic material, it is basic to take it as cementing material due to the presence of SiO₂, Al₂O₃, Fe₂O₃, and CaO.

2.1.3. Fine Aggregates. Fine and coarse aggregates used in the study were obtained from quarries near Debre Tabor, Ethiopia. According to ASTM C33, aggregates that are less than 4.75 mm are designated as fine aggregate. For this study, locally available well-graded river sand free from deleterious materials is used as a fine aggregate. Several tests are carried out to investigate the quality of the fine aggregate in accordance with the ASTM standard. The sand used in the study has the following properties that meet ASTM standards as shown in Table 2 and Figure 2.

2.1.4. Coarse Aggregates. Well-graded crushed basaltic stone coarse aggregate was used and washed to make it free from dust and deleterious materials, which meet the requirements of ASTM standards. Table 3 and Figure 3 show the properties and gradation of coarse aggregate.
2.2. Mix Design. The ACI 211.1 method was used in this study to specify the quantities and proportions of concrete. The study was conducted by using concrete with a compressive strength of 25 MPa. Five concrete mixes were prepared for different percentages of CHA from 0% to 20% by weight of cement with 5% increment which are formulated by trial and error. A constant water-cement ratio of 0.5 and a slump range of 25–75 mm was used for all concrete mixtures. The reference concrete mix proportion is shown in Table 4.

2.3. Test Methods. The engineering property of all materials necessary for describing the type of materials used and properties that can affect the production of concrete was determined before production. All ingredients of concrete mixes are measured by weight according to their proportions; the mixing was done using a pan concrete mixer as per ASTM C-192. To test the workability of concrete, a slump test was made by slump cone and fed directly into the cube molds and compacted by hand for slumps less than 75 mm and then demolded after 24 hrs of casting and immersed into curing tank.

After the various tests done on the materials properties of cement, fine aggregates, coarse aggregates, and CHA, the fresh property of all concrete samples identified the workability of the concrete. To check the workability of concrete, the slump test was carried out in the fresh concrete mix with ASTM C143. The consistency and setting times of CHA and OPC was carried out with ASTM C-187 and ASTM C-191.

For the mechanical properties of concrete tests, a total of 147 (150 mm × 150 mm × 150 mm) concrete cube specimens were prepared. The prepared samples were demolded after 24 hrs and immersed to water for curing. Then, compressive strength test was conducted as per ASTM C-109 at different curing ages (3, 7, 28, 56, and 91 days).

Sulphate resistance of concrete was conducted as per ASTM C-1012. Samples with 150 mm×150 mm×150 mm dimension were prepared and soaked in 5% Na$_2$SO$_4$ solution, and compressive strength of samples were tested on after 28, 56, and 91 days of curing. Water absorption tests of concrete mixtures were studied as per ASTM C-642 with different replacement level of CHA.

The microstructure of the concrete was determined by FTIR and TGA/DTA methods. Samples were collected from inner parts of compressive strength tested mixtures. Then, the samples were immersed in 97% ethanol solution for 3 days to stop hydration. After 3 days, samples were oven-dried for 24 hours at 105°C+5°C and grounded. The grounded samples were sieved, and samples passed through using 75 μm sieve were used for microstructure studies.

The data analysis was made with a series of steps. The first step is to analyze the chemical composition of CHA, which is conducted at the Ethiopian Geological Survey laboratory, and then the workability, compressive strength, and water absorption of concrete were discussed and analyzed, which includes concrete immersed in sulphate solution. The second step was to analyze microstructure of concrete result from FTIR, and TGA/DTA and ORIGIN PRO-2021 were used to draw the basic graphs and to correlate the results using linear relation. Finally, conclusion was drawn.

3. Results and Discussion

3.1. CHA on the Concrete Fresh Properties

3.1.1. Consistency. The normal consistencies of pastes containing CHA are shown in Figure 4. The control paste had a normal consistency of 27%. All of the pastes containing coffee husk ash shows normal consistency higher than the control paste, CHA 0%. Up to 15% replacement level, the normal consistency was within the range of 27%–33% that is specified by ASTM C-187, while at 20% replacement, the normal consistency exceeds from the maximum consistency, and a consistency of 35% was recorded.

In general, the consistency of blended cement showed an increasing trend with a rise in CHA content. This happens because an increase in CHA content will increase the number of fine particles in the mix that subsequently increase the demand of mixing water.

3.1.2. Setting Times. The initial and final setting times of mixtures with different CHA content are displayed in Figure 5. The figure revealed that the initial and final setting times were in the range of 67–126 minutes and 310–524 minutes, respectively, and satisfy the requirements of ASTM C-191.

In general, the setting time of concrete showed an increasing trend with an increase in share of CHA content due to the fact that primary cement hydration will be decreased with the reduction of cement content. The increase in CHA content significantly reduces hydration of blended cement as CHA has very low amount of CaO compared to cement. Consequently, CHA will possess secondary reaction which ultimately increases the setting times of the blended cement. However, CHA replacement can satisfy setting time requirement of the blended mix up to 20% CHA replacement.
3.1.3. Workability. The summary of the slump test results of mixtures is presented in Figure 6. As shown in the figure, the slump of concrete was measured between 15mm and 35mm. In general, the slump of concrete showed a decreasing trend with an increase in CHA content. This may be due to CHA has a high specific surface area and increases the water demand of the concrete mixture to produce workable concrete. CHA has finer particles, which have a higher surface area compared to cement particles. The water-absorbing characteristics of CHA increase the demand for water with the increasing amount of CHA in the mixture [1].

3.2. CHA on the Concrete Mechanical Properties

3.2.1. Compressive Strength. Figure 7 shows the compressive strength results of mixes in pure water (Figure 7). The densification of gel decreased with increasing in CHA content, and this will reduce the ability of concrete to withstand sulphate penetration [28]. The sulphate attack effect significantly increased with increasing curing ages, and the maximum compressive loss was measured at 91 days.

As shown in Figure 8, the compressive strength loss of concrete under sulphate solution increased with increasing CHA content. This result is in good agreement with compressive strength of mixes in pure water (Figure 7). The densification of gel decreased with increasing in CHA content, and this will reduce the ability of concrete to withstand sulphate penetration [28]. The sulphate attack effect significantly increased with increasing curing ages, and the maximum compressive loss was measured at 91 days.

3.3. CHA on the Concrete Mechanical Properties

3.3.1. Sulphate Attack of Concrete. The purpose of the sulphate resistance test was to determine the sulphate resistance of CHA concrete. The concrete after demolding was immersed in 5% Na₂SO₄ solution, and then the compressive strength of the immersed concrete was checked as followed by ASTM C-109 for 0%–10% replacement at 28, 56, and 91 days of curing.

As shown in Figure 8, the compressive strength of mixes in pure water (Figure 7). The densification of gel decreased with increasing in CHA content, and this will reduce the ability of concrete to withstand sulphate penetration [28]. The sulphate attack effect significantly increased with increasing curing ages, and the maximum compressive loss was measured at 91 days.

3.3.2. Water Absorption. ASTM C-140 laboratory test was conducted to determine the water absorption of concrete. Figure 9 shows the water absorption of concrete mixes in different curing ages.

The water absorption of mixes was measured between 3% and 13.83%. In general, the water absorption results increased as the portion of CHA content increases in the mixes which is in good agreement with compressive strength of concrete. The increase in CHA content will decrease the densification of CSH gels which eventually make fragmented and porous morphology. This ultimately leads to a higher water absorption of mixes with increasing CHA content [29].

3.3.3. Relationship between Compressive Strength and Water Absorption. To investigate the relationship between the compressive strength and water absorption of CHA concrete, linear regression was developed. As expected, water absorption and compressive strength were inversely correlated, following the equations $y = -2.5325x + 57.098$ with an $R^2 = 0.8652$.

As shown on Figure 10, the compressive strength is increasing as the curing days increase, but the water absorption is decreasing. This is because when the concrete age...
is increasing, the concrete is becoming dense and its water absorption capacity is decreasing. In addition, compressive strength is inversely proportional to water absorption since the water absorption is increasing as the CHA content is increasing. This is due to the surface area is increasing as the CHA replacement is increasing [29].

3.4. CHA on the Concrete Microstructure Characteristics. The microstructure of CHA is depending on the processing methods [2]. The microstructural investigation of the CHA concrete sample pastes using thermal analysis methods (FTIR and DTA/TGA) is presented in a graph, and the analysis was done based on the graphs presented.

3.4.1. FTIR Analysis. From Figure 11, the FTIR analysis was done for the concrete paste taken from the compressive test by taking the mortar part and immersing it using 97% ethanol for three consecutive days to stop the hydration reaction, and then oven-dried for 24 hours; finally, crush it until it passes 75 μm sieve. The thermal investigation done for 0%, 5%, 10%, and 15% samples at 28, 56, and 91 days of curing. The vibration modes were compared with the spectra available in the literature. Figure 11 shows the infrared spectrum of all samples at 28 curing days with characteristic wavenumber from 500 to 4000 cm⁻¹ and the associated functional groups of mixtures. From the result of the FTIR spectrum analysis of CHA, based on the findings [30, 31], undissolved or unreacted CHA, particles are traced at 431, 491, 485, 450, 885, 883, 868, and 877 at FTIR spectra of selected samples after 28 days of curing.

Water bond (O-H stretching and bending) related with the hydrated gels of CHA-OPC reaction was exhibited in CHA samples at a broad absorption band of around 3460 cm⁻¹ and in a weak band around 1650 cm⁻¹. The intensity of the O-H broadband of CHA-15% appeared to be higher than CHA-0% as the higher fineness CHA increased gel formation [31, 32].

Similar results were reported in the research findings [31]. The main band for CHA C-S-H gel was created at about 982, 992, 995, and 1008 cm⁻¹ for CHA 0%, 5%, 10%, and 15%, respectively.

In addition, from the FTIR graphs (Figure 11) at CHA-10% replacement level, the curve becomes wide, and hence, a higher C-S-H gel was formed, and hence, the compressive strength becomes optimum at this point [18, 24, 32].

<table>
<thead>
<tr>
<th>Mix code</th>
<th>Per CHA (kg)</th>
<th>Cement (kg)</th>
<th>Water (lit)</th>
<th>Coarse aggregate (kg)</th>
<th>Sand (kg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CHA-0%</td>
<td>m³</td>
<td>0</td>
<td>358</td>
<td>186.3</td>
<td>1135.3</td>
</tr>
<tr>
<td>Trial</td>
<td>0</td>
<td>4.9</td>
<td>2.45</td>
<td>14.9</td>
<td>11.7</td>
</tr>
</tbody>
</table>

Table 3: Properties of coarse aggregate.
results obtained from C-S-H gel formation are related to the compressive strength results. Carbonates (C-O-C) and undissolved compounds are located approximately 1450 cm$^{-1}$ [31].

3.4.2. TGA/DTA Analysis. The TGA/DTA simultaneous thermal analysis was used to evaluate the thermal stability of the concrete sample after replacing CHA with four types of replacement proportions (0%, 5%, 10%, and 15%) for 28, 56,
and 91 days of curing. TGA/DTA materials analysis highlighted their thermal stability and compound content/type by plotting two curves based on temperature at the same time.

As stated by other findings [34], from 153°C–483°C and 483°C–700°C indicates the presence of organic matter and calcite. The combustion of the organic matter from the above ranges and the thermal decomposition of calcite (CaCO₃) occur at about 500°C. From the TGA curve, it is observed that the first mass loss is evident between 70°C and 200°C with a mass loss of 4.83, 5.25, 4.83, and 4.3%, respectively, for each CHA content. Similarly, the second mass loss is observed between 490°C and 742°C with a mass loss of approximately 8% for each replacement level.

As we can see from the DTA graph, there are four peaks at 474, 514, 696, and 773°C; 464, 500, 661, and 756°C; 480, 519, 693, and 778°C; 465, 500, 689, and 753°C, respectively, for 0%–15% replacement levels. For each peak level, there is a loss in weight of the sample with the rate of the heat of about 2–4°C/min. These peaks correspond to the removal of
water molecules, which are free or bound with the structural compounds [35].

Similarly, from the TGA (Figure 12) and DTA (Figures 13 and 14) graph, approximately at 500°C, the carbonation of the sample was carried out, and hence, it results in the weight/mass of the sample. The sample was fully carbonized at 500°C.

From the DTA graph (Figure 13), the loss of free water through a process of evaporation and dehydration of CSH gel appeared at about 150°C. Moreover, decomposition of ettringite occurred at about 500°C, whereas at 720°C, decomposition of calcite material is appeared to happen. Similar findings were reported by pervious research studies [36].

Hence, the TGA/DTA results show that the C-S-H gel would be in the range of 950–1100 cm$^{-1}$ wavenumber and at 500°C, respectively, and the CHA sample was decomposed and mass loss was observed at 500°C.

4. Conclusions

In this research, the use of coffee husk ash as a cement replacing material in concrete production is studied, and upon the investigation results, the following conclusions are made:

(i) The use of CHA in concrete reduces the workability of concrete as the content of CHA increased in all samples. Yet, sufficiently workable concrete was prepared up to 20% CHA replacement level.

(ii) The compressive strength of concrete decreased with increasing CHA content. Even though the compressive strength result showed a decreasing trend, 5% CHA and 10% CHA mixes meet the designed strength of concrete after 28 curing days.

(iii) The water absorption is increasing as the CHA content is increasing, but it decreases as the curing day increases.

(iv) The sulphate attack of concrete increases with increasing CHA content and increasing curing ages.

(v) The FTIR and TGA/DTA results show that the C-S-H gel would be in the range of 950–1100 cm$^{-1}$ wavenumber and at 500°C, respectively, and the CHA sample was decomposed and mass loss was observed at 500°C.

In general, from the compressive strength, the concrete prepared with CHA has attained its design strength up to 10% replacement level cement. Hence, CHA has great potential to partially replace cement up to 10% by weight of cement in concrete production whereby reducing the environmental effect of cement production.

In addition, the study greatly recommended further investigation on the long-term effects of CHA containing concrete to find out the relevant other durability properties of the concrete. [33].

Data Availability

Data are available from the corresponding author upon request.

Conflicts of Interest

The authors declare that there are no conflicts of interest regarding the research authorship and publication of this article.

Authors’ Contributions

Asmamaw Gedefaw conducted the experiment and drafted the manuscript. Begashaw Worku revised the manuscript. Solomon Asrat Endale drafted and revised the manuscript. Betelhem Tilahun Habtegebreal Conducted the experiment. Mitiku Damtie Yehualaw supervised the experiment and drafted and revised the manuscript.

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