Pozzolanic Properties of Agro Waste Ashes for Potential Cement Replacement Predicted using ANN

Manikanta C1, Manikandan P1, Duraimurugan S2, Elavenil S1, Vasugi V1,3
1School of Civil Engineering, VIT Chennai, India
2Fosroc Chemicals India Pvt. Ltd., Chennai, India
3Email: vasugi.v@vit.ac.in

Abstract: Every year, cement demand increases due to infrastructural development in our country, which indirectly increases global warming since the manufacturing of Portland cement emits a large volume of carbon dioxide into the atmosphere. Hence, the demand of using Portland cement should be reduced by using various types of mineral admixtures as a replacement to Cement. In India, every year agricultural sector produces a large volume of waste by-products like corn cob, rice husk, sugarcane bagasse, bamboo leaves etc., which could be burnt in a controlled environment and the resulted ashes could be used as partial replacement to Cement. The pozzolanic activity of agro-waste ashes like rice husk ash, cocoa fruit shell ash, corn cob ash, bamboo leaves ash, sugarcane bagasse ash have been tested and compared using the strength activity test, the saturated lime test and the Frattini test with the implementation of Artificial Intelligence using Neural Network was carried out. It is observed that Rice husk ash, corn cob ash, bamboo leaf ash, sugar cane bagasse ash have the potential to be replaced for Cement while cocoa fruit shell ash does not have the expected pozzolanic activity. The predicted pozzolanic strength property of the different agro-waste ashes with the utilization of Artificial Neural Network was observed to correspond with experimental values.

Keywords: Agro waste ashes, Frattini test, Strength activity Index, XRD, ANN.

1. Introduction
The usage of cement concrete is ever increasing due to infrastructural development and urbanization in our country. However, the manufacturing of Cement results in global warming by releasing more carbon dioxide in the atmosphere. It is mandated to find ways and means to reduce the demand for Cement by replacing with some other secondary cementitious materials [1]. Industrial by-products like silica fume, Slag, metakaolin, fly ash, pond ash etc., have been reported as secondary cementitious materials [2-4]. Still, search for more such secondary cementitious materials from different resources is continuing. India is an agricultural country, every year our country produces many waste by-products from agriculture like rice husk, corn cob, sugarcane bagasse, bamboo leaves, cocoa fruit shells, coconut shells, wheat husk etc.

In this experimental work, Rice husk ash (RHA), Corncob ash (CCA), Sugarcane bagasse ash (SBA), Bamboo leaf ash (BLA) and cocoa fruit shell ash (CFSA) has been taken to test the potential replacement of Cement as per pertinent codes. RHA: Nearly 120 million tons of paddies are produced in India every year. Rice husk means hard protecting the cover of rice grains. About 20-25% of rice husk is generated from paddy during milling of paddy grains, and 20% of total husk becomes ash [5].

Corncob Ash: the annual production of corn is about 25 million tons in India [6]. Corncob is a waste product obtained during milling for corn gains. Every tonne of corn will produce 200kgs of the corncob [7]. Sugarcane bagasse Ash: Uttar Pradesh, Maharashtra and Karnataka states are high cultivators of sugarcane in India. Sugarcane bagasse is an outcome waste product from the factory during the production of sugar [8]. Every one tonne of sugarcane will produce 300 kilograms of bagasse waste [9]. Bamboo leaves Ash: Bamboo trees are beneficial for making many things. Large stems are used to build small houses and small bridges and other things. Bamboo is a
natural construction material, and it is not expensive. Every year 20 million tons of bamboo leaves produce in the world [10]. In that, nearly 6 million tons produced by India. Cocoa fruit shell Ash Andhra Pradesh had the highest volume of cocoa fruit production in India. Cocoa grains highly used in the preparation of chocolates and vegetable oils. Cocoa fruit shell means the outer layer of cocoa fruits. These agricultural wastes have been collected, dried and controlled burnt in an environmental chamber. Pozzolanic activity of five different test pozzolans (agro- waste ashes) was tested with four different pozzolanic activity test methods to identify the potential replacement of Cement microstructure were studied with XRD.

ANN is a mathematical and numerical tool that functions as a set of neurons that exists in the human brain. It could be widely employed in the field of engineering applications to resolve extremely complex solution. Recent research articles proved that ANN model could be effectively applied in construction materials to predict the essential factors like the content of Cement [9], mix concrete design [12], drying shrinkage property of concrete [13], percentage replacement amount of recycled aggregates [14], workability of concrete, compressive strength [15] and mechanical strength properties [16] with more accuracy. Further, the ANN model was framed with the Lavenberg Marquardt (LM) algorithm in MATLAB to predict the pozzolanic strength properties of different agro-based ashes.

2. Materials and Methods

2.1. Materials

The agro wastes Viz., RHA, CCA, BLA, SBA, and CFSA were obtained by controlled burning in the Laboratory oven at 900°C for 15 minutes. The specific gravity and % fineness obtained are shown in Table1. The mix proportions recommended as per pertinent codes are given in Table 2.

| Physical Property | R.H.A | C.C.A | B.L.A | S.B.A | C.F.S.A |
|-------------------|-------|-------|-------|-------|---------|
| Specific gravity  | 2.16  | 2.51  | 2.32  | 2.24  | 2.46    |
| Fineness (%)      | 1.87  | 2.32  | 2.67  | 2.71  | 2.25    |

Table 2. Details of Mix proportions for the preparation of test samples.

| Test/material | P.C | Sand | Pozzolan | Water | Ca(OH)2 | No. of samples |
|---------------|-----|------|----------|-------|---------|----------------|
| S.A.I*/control| 450 | 1350 | 0        | 2     | -       | 6              |
| S.A.I/R.H.A   | 360 | 1350 | 9        | 2     | -       | 6              |
| S.A.I/C.C.A   | 360 | 1350 | 9        | 3     | -       | 6              |
| S.A.I/B.L.A   | 360 | 1350 | 9        | 2     | -       | 6              |
| S.A.I/S.B.A   | 360 | 1350 | 9        | 3     | -       | 6              |
| S.AI/C.F.S.A  | 360 | 1350 | 9        | 2     | -       | 6              |
| F.T*/control  | 2   | -    | 0        | 1     | -       | 1              |
| F.T/R.H.A     | 1   | -    | 4        | 1     | -       | 1              |
| F.T/C.C.A     | 1   | -    | 4        | 1     | -       | 1              |
| F.T/B.L.A     | 1   | -    | 4        | 1     | -       | 1              |
| F.T/S.B.A     | 1   | -    | 4        | 1     | -       | 1              |
| F.T/C.F.S.A   | 1   | -    | 4        | 1     | -       | 1              |
| S.L.T*/control| -   | -    | 0        | -     | 7       | 3              |
2.2. Pozzolanic activity test methods

2.2.1. Strength Activity Index
Strength Activity Index (SAI) is based on code BS 3892, 1997. Test samples were prepared by mixing 1350g sand, 450g cement & 225 ml water in a mixer for 10 minutes. Test samples were prepared 20% cement preplaced with test pozzolan. Then flow tests were carried out on that pastes for finding water-cement ratio based on code IS: 5515-1969. After that mortar paste was remixed, it is then cast into cube molds using table vibrator. Cube moulds were demoulded after 24 hours and placed in the water tank up to the test date.

\[
\text{Strength activity index (SAI)} = \frac{A}{B} \times 100
\]

where

- \(A\) – Compressive strength of test pozzolan Sample (MPa).
- \(B\) – Compressive strength of control mortar Sample (MPa).

2.2.2. Frattini Test
Frattini Test is based on code EN 196-5, 2005 [17]. The control samples were prepared by mixing 20g cement & 100 ml water. Then test samples were prepared by 20% cement preplaced with test pozzolan. After that, samples were left for 8days in a plastic bottle in an oven at 40°C. After eight days, samples were filtered with filter paper (Whatman No.542) and tested as recommended in the code.

2.2.3. Saturated Lime Test
In the Saturated lime test, samples were prepared with 1g pozzolan mixed with a 75ml saturated lime solution. After that, test samples were left for 7, 14, 28 days in a plastic bottle in an oven at 40°C and then tested as per code.

2.2.4. Lime Reactivity Test
Lime Reactivity Test is based on IS: 1727-1967 [18]. Test samples were prepared by mixing 150gms Hydrated lime with 1350gms of standard sand and 300 × M (specific gravity of pozzolan/specific gravity of lime) grams. Then flow tests were carried out on that paste for finding water-cement ratio. After that, mortar paste were remixed & cast into cube molds by using a vibrating table. Cube moulds were covered with a smooth and greased glass plate then transferred to wet gunny bags for 48 hours after that demoulded cube specimen were cured at 90 to 100 % humidity up to test date.

| S.L.T/R.H.A | - | - | 1 | - | 7 | 3 |
|-------------|---|---|---|---|---|---|
| S.L.T /C.C.A| - | - | 1 | - | 7 | 3 |
| S.L.T /B.L.A| - | - | 1 | - | 7 | 3 |
| S.L.T /S.B.A| - | - | 1 | - | 7 | 3 |
| S.L.T/C.F.S.A| - | - | 1 | - | 7 | 3 |
| L.R.T\(^a\)/control| - | 1350 | 3 | 3 | 1 | 9 |
| L.R.T/R.H.A | - | 1350 | 3 | 5 | 1 | 9 |
| L.R.T/C.C.A | - | 1350 | 3 | 5 | 1 | 9 |
| L.R.T/B.L.A | - | 1350 | 3 | 5 | 1 | 9 |
| L.R.T/S.B.A | - | 1350 | 3 | 5 | 1 | 9 |
| L.R.T/C.F.S.A | - | 1350 | 3 | 5 | 1 | 9 |

\(^{a}\)Strength Activity Index,  
\(^{b}\)Frattini Test,  
\(^{c}\)Saturated Lime Test,  
\(^{d}\)Lime Reactivity Test
2.3. Prediction Of pozzolanic strength properties of agro-waste ashes using ANN framework

ANN framework can be defined as the combination of hardware and software which are interlinked like the structure of the neurons present in the human brain. ANN can be classified under the category of deep learning technique, which is employed for predicting the results where no formula is required between input and output variable [19]. The ANN framework comprises five major components: input variables, weights, sum function, activation function, and output variables. ANN comprises many interlocked neuron-like structures in which each of them generates output from the various input data’s as mentioned in equation 1 [20].

\[ Y = F \left( \sum_{j=0}^{n} X_j W_j - B \right) \]  

(1)

Where, ‘X<sub>j</sub>’ represents the input of the j<sup>th</sup> neuron, ‘W<sub>j</sub>’ indicates the weight of the j<sup>th</sup> neuron, ‘F’ denotes the activation function, ‘n’ is the number of neurons present, ‘Y’ is the output and ‘B’ is the bias. The term \( \sum_{j=0}^{n} X_j W_j - B \) exists in equation (1) denotes the input function of the activation function (F). Basically, non-linear activation functions like sigmoidal steps are utilized in ANN, represented in equation (2).

\[ F(t) = \frac{1}{1 + e^{-\alpha t}} \]

where \( t = \sum_{j=0}^{n} X_j W_j - B \)  

(2)

In equation 2, ‘\( \alpha \)’ indicates the constant term used to regulate the gradient in the non-linear phase.

ANN comprises three layers; therefore, it can be stated as a Multilayer perception model as depicted in the figure 1. The first layer is termed as an input layer, which consists of 5 different variables (PC content, quantity of sand, quantity of cement, water content and Ca(OH)<sub>2</sub> levels). The subsequent layer is designated as a computational layer or hidden layer, and the last layer is the output layer, where the ANN model predicts the pozzolanic strength properties of various agro-waste ashes. The input variables assigned for the selected ANN framework were designated as PC content (X<sub>1</sub>), quantity of sand (X<sub>2</sub>), pozzolan content (X<sub>3</sub>), amount of water added (X<sub>4</sub>) and Ca(OH)<sub>2</sub> level (X<sub>5</sub>). Similarly, the output variable selected is pozzolanic strength properties of different agro-based waste ashes such as strength activity index, percentage of CaO removed from the Frattini test, percentage of CaO removed from saturated lime test and strength compressive strength results obtained from lime reactivity test (Y<sub>1</sub>).

In this study, a three-layer perception ANN framework was selected in MATLAB (2016) using the Lavenberg-Marquardt (LM) algorithm with a feed-forward back propagation technique for predicting the different pozzolanic strength properties of five different agro-based by-products [21]. The complete number of computational layers and the exact number of neurons present in each computational layer in the ANN model can be decided by conducting series of trials at the time of training and testing phase until expected results are obtained. For the present study, ANN architecture comprising two computational layers with five numbers of neurons in every layer was selected for predicting the pozzolanic strength properties. The optimization of the output results obtained from ANN architecture is achieved using the Mean Square Error (MSE) method between targeted and predicted values is given in equation 3 [22].
\[ \text{MSE} = \frac{1}{2} \sum_{j=1}^{n} (\bar{y}_j - y_j)^2 \]  

Where, \( n \) represents the number of values, \( \bar{y} \) is the experimental values, and \( y \) is the predicted values. The details of the input and output values considered for the present ANN model are presented in Table 3.

**Table 3:** Input and Target variables accounted for the ANN study.

| Input and the Target variable | Range     | Remarks        |
|------------------------------|-----------|----------------|
| Water content (ml)          | 100 – 560 | Input Variables|
| PC content (g)              | 16 – 450  |                |
| Sand (g)                    | 1350      |                |
| Pozzolan (g)                | 0 – 377   |                |
| Ca(OH)\text{$_2$} level (ml) | 75 – 150  |                |
| SAI                         | 47.2 – 100| Output Variables|
| Frattini test               | 5.2 – 75.8|                |
| Saturated Lime Test         | 9.1 – 70.4|                |
| Lime Reactivity Test        | 0 – 16.2  |                |

3. Results and Discussion

3.1 Strength Activity Index

The 7 & 28 days compressive strength of the test samples are given in Table 4. It is observed that RHA, CCA, BLA have shown with more than 75% strength activity index while SBA and CFSA resulted in less than 75% strength activity index at 28days, which should be considered for potential replacement of cement.
Table 4. Compressive strength of samples for 7 days & 28 days.

| S.I.No | Materials | 7 day Compressive strength (MPa) | SAI (%) | 7 day Compressive strength (MPa) | SAI (%) |
|-------|-----------|----------------------------------|---------|----------------------------------|---------|
| 1.    | Control   | 34.08                            | 100     | 46.3                             | 100     |
| 2.    | RHA       | 22.61                            | 66.34   | 40.5                             | 87.04   |
| 3.    | CCA       | 20.08                            | 58.92   | 36                               | 77.61   |
| 4.    | BLA       | 26.80                            | 78.52   | 41.9                             | 90.49   |
| 5.    | SBA       | 18.80                            | 55.16   | 32                               | 69.11   |
| 6.    | CFSA      | 16.10                            | 47.2    | 29                               | 62.63   |

Figure 2 demonstrates that bamboo leaves ash (BLA) and Rich husk ash (RHA) show a good pozzolanic activity of 90.49% & 87.04%, respectively. Cocoa fruit shell ash is less pozzolanic (60%) compared to other agro waste ashes taken for tests.

![Figure 2. Variation of Strength Activity Index.](image)

3.2 Frattini Test

The reduction of CaO is more in RHA and BLA and SBA, revealing that these agro-waste ashes are pozzolanic (Table 5) and can replace the Cement to some extent.

Table 5. CaO reduction in Frattini test.

| S.I.No | Material | (OH) mmol l^{-1} | (CaO) mmol l^{-1} | Theoretical Max. (CaO) mmol l^{-1} | (CaO) Reduction (%) |
|--------|----------|------------------|-------------------|------------------------------------|---------------------|
| 1.     | Control  | 58.1             | 7.7               | 8.1                                | 5.2                 |
| 2.     | RHA      | 39.8             | 3.4               | 14.1                               | 75.8                |
| 3.     | CCA      | 52.0             | 4.6               | 9.4                                | 51.1                |
| 4.     | BLA      | 44.1             | 3.5               | 12.0                               | 70.9                |
| 5.     | SBA      | 42.6             | 4.1               | 12.6                               | 67.6                |
| 6.     | CFSA     | 50.2             | 4.3               | 9.92                               | 56.7                |

3.3 Saturated Lime Test

Like the Frattini test, saturated lime test revealed the total CaO removed at 7, 14, and 28 days, as shown in Table 6. RHA and SBA have shown more pozzolanic activity compared with other agro waste ashes considered. Variation of total CaO removal (%) is shown in Figure 3. It is observed
that the total CaO removal is in the range of 60-70% at 28 days in all the agro-waste ashes considered.

Table 6. Saturation lime test results for samples.

| S.I.No | Material | 7 days (CaO) removed (%) | 14 days (CaO) removed (%) | 28 days (CaO) removed (%) |
|--------|----------|--------------------------|---------------------------|---------------------------|
| 1      | Control  | 9.1                      | 13.2                      | 17.1                      |
| 2      | RHA      | 60.9                     | 65.5                      | 70.4                      |
| 3      | CCA      | 45.8                     | 51.6                      | 59.2                      |
| 4      | BLA      | 47.2                     | 51.2                      | 63.3                      |
| 5      | SBA      | 45.2                     | 54.3                      | 68.1                      |

Figure 3. Variation of total CaO removed at 7, 14 & 28 days.

3.4 Lime Reactivity Test

Similar to Strength activity index tests, the Lime reactivity test reveals pozzolanic property in terms of compressive strength. Table 7 depicts the 7, 14 and 28 days compressive strength for all the five agro-waste ashes considered. Lime reactivity test results show low strength at 7 days, but strength increased after 28 days, indicating that the pozzolanic reactions are slow but improve the secondary CSH gels in the cementitious matrix. Based on these results from all the four tests considered, RHA and BLA have more pozzolanic properties.

Table 7. Compressive strength at 7, 14 and 28 days.

| S.I.No | Material | 7-day (Compressive strength) (MPa) | 14-day (Compressive strength) (MPa) | 28-day (Compressive strength) (MPa) |
|--------|----------|-----------------------------------|-------------------------------------|-------------------------------------|
| 1      | control  | 5.2                               | 8.8                                 | 16.2                                |
| 2      | RHA      | 4.1                               | 7.1                                 | 15.2                                |
| 3      | CCA      | 3.2                               | 5.4                                 | 11.8                                |
| 4      | BLA      | 4.8                               | 8.2                                 | 15.9                                |
| 5      | SBA      | 4.0                               | 6.1                                 | 10.6                                |
3.5 Predicted Pozzolanic strength properties from ANN model

The actual, predicted, and the ratio of actual/predicted pozzolanic strength results obtained from the Strength Activity Index test, Frattini test, saturated lime test and Lime reactivity test for various agro- based ashes are listed in Tables 8-11. From the above tables, it was observed that the experimental and ANN models' results are comparable. The percentage error observed from MSE for strength activity index, the proportion of CaO removed in the Frattini test, percentage of CaO removed from the saturated lime test, and compressive strength results obtained from the lime reactivity test are 1.3%, 2.4%, 1.93 % and 5.34 % respectively. The error values acquired from the MSE can be minimal as it lies with 10%. From the above discussions, it can be inferred that the ANN model can be employed efficiently in predicting the pozzolanic strength properties of corn cob, rice husk, sugarcane bagasse and bamboo leaf ashes.

Table 8. Experimental and Predicted results of Strength Activity Index.

| Mix ID       | SAI (%) at 7 days Exp | Pred | Exp/Pred | SAI (%) at 28 days Exp | Pred | Exp/Pred |
|--------------|-----------------------|------|----------|------------------------|------|----------|
| SAI/Control  | 100                   |      | 1.01     | 100                    |      | 1.01     |
| S.A.I/R.H.A  | 66.34                 | 68.25| 0.97     | 87.04                 | 70.21| 1.24     |
| S.A.I/C.C.A  | 58.92                 | 60.23| 0.98     | 77.61                 | 57.32| 1.35     |
| S.A.I/B.L.A  | 78.52                 | 79.97| 0.98     | 90.49                 | 76.39| 1.18     |
| S.A.I/S.B.A  | 55.16                 | 57.29| 0.96     | 69.11                 | 59.21| 1.17     |
| SAI/C.F.S.A  | 47.2                  | 45.92| 1.03     | 62.63                 | 42.34| 1.48     |

Table 9. Experimental and Predicted results obtained from Frattini test.

| Mix ID       | CaO reduction in Frattini test (%) Exp | Pred | Exp/Pred |
|--------------|---------------------------------------|------|----------|
| FT/control   | 5.2                                   | 4.93 | 1.05     |
| F.T/R.H.A    | 75.8                                  | 77.3 | 0.98     |
| F.T/C.C.A    | 51.1                                  | 48.23| 1.06     |
| F.T/B.L.A    | 70.9                                  | 70.12| 1.01     |
| F.T/S.B.A    | 67.6                                  | 67.93| 1.00     |
| F.T/C.F.S.A  | 56.7                                  | 54.23| 1.05     |

Table 10. Experimental and Predicted results of Strength Activity Index.

| Mix ID       | CaO reduction at 7 days (%) Exp | Pred | Exp/Pred | CaO reduction at 14 days (%) Exp | Pred | Exp/Pred | CaO reduction at 28 days (%) Exp | Pred | Exp/Pred |
|--------------|---------------------------------|------|----------|---------------------------------|------|----------|---------------------------------|------|----------|
| SL/control   | 9.1                             | 10.23| 0.89     | 13.2                            | 10.94| 1.21     | 17.1                            | 15.43| 1.11     |
| S.L/R.H.A    | 60.9                            | 63.23| 0.96     | 65.5                            | 65.97| 0.99     | 70.4                            | 69.43| 1.01     |
| S.L/C.C.A    | 45.8                            | 53.54| 0.86     | 51.6                            | 50.32| 1.03     | 59.2                            | 56.43| 1.05     |
| S.L/B.L.A    | 47.2                            | 45.12| 1.05     | 51.2                            | 49.75| 1.03     | 63.3                            | 61.23| 1.03     |
| S.L/S.B.A    | 45.2                            | 45.7 | 0.99     | 54.3                            | 53.21| 1.02     | 68.1                            | 66.24| 1.03     |
| S.L/C.F.S.A  | 49.1                            | 48.32| 1.02     | 53.1                            | 55.56| 0.96     | 58.4                            | 55.43| 1.05     |
Table 11. Comparison of Experimental and Predicted strength results from Lime reactivity test.

| Mix ID       | Compressive strength at 7 days (MPa) | Compressive strength at 14 days (MPa) | Compressive strength at 28 days (MPa) |
|--------------|-------------------------------------|--------------------------------------|--------------------------------------|
|              | Exp Pred | Exp/Pred | Exp Pred | Exp/Pred | Exp Pred | Exp Pred | Exp/Pred |
| L.R.T/co     | 5.2      | 4.97     | 1.05     | 8.8      | 9.53     | 0.92     | 16.2      | 15.21    | 1.07     |
| L.R.T/R      | 4.1      | 4.36     | 0.94     | 7.1      | 8.21     | 0.86     | 15.2      | 17.61    | 0.86     |
| L.R.T/C      | 3.2      | 2.74     | 1.17     | 5.4      | 6.43     | 0.84     | 11.8      | 13.2     | 0.89     |
| L.R.T/B      | 4.8      | 5.34     | 0.90     | 8.2      | 7.9      | 1.04     | 15.9      | 17.63    | 0.90     |
| L.R.T/S      | 4.4      | 4.85     | 0.88     | 6.1      | 5.89     | 1.04     | 10.6      | 11.23    | 0.94     |
|              | 0        | 0        | 0        | 0        | 0        | 0        | 0         | 0        | 0        |

4. Characterization with XRD

All the agro-waste ashes viz., RHA, CCB, BLA, SBA and CFSA were characterized with XRD analysis and the respective XRD patterns are shown in Figures 4 to 8. Except for RHA, the other four agro-waste ashes considered for this study shows more amorphous phases. It is also observed that Quartz is present in all the agro-waste considered in a significant amount. Quartz is the main crystalline phase present in all ashes. In RHA, Quartz shows a high intensity of up to 5000 arbitrary units while in CFSA, Quartz shows a low intensity of 450 arbitrary units. CCA, BLA and SBA Shows between 1200 to 1400 arbitrary units.

![Figure 4. XRD of Rice husk ash.](image1)

![Figure 5. XRD of Corncob ash.](image2)

![Figure 6. XRD of Bamboo leaves ash.](image3)

![Figure 7. XRD of Sugarcane bagasse ash.](image4)
5. Conclusion
Based on the test conducted and the results obtained, the following conclusions were drawn.
- Bamboo leaf ash and Rice husk ash resulted in 90.49 % & 87.04 % strength activity index show a potential cement replacement.
- Frattini test & saturated lime test results show that Agro waste ashes remove a high percentage of CaO compared with Cement.
- Rice husk ash, Bamboo leaf ash observed to be more pozzolanic when compared with Corncob ash and sugarcane bagasse ash.
- The application of the ANN framework, which primarily works on the experimental results, turned out to be one of the valuable tools to predict the pozzolanic strength properties of different agro based ashes.

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