## **TECHNICAL PAPER**



# **Impact of nano ZnO particles on the characteristics of the cement mortar**

**Hiteshkumar Patil[1](http://orcid.org/0000-0002-6850-2336) · Arunkumar Dwivedi<sup>2</sup>**

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## **Abstract**

In practice, it is very common to estimate the strength of concrete by destructive or by partial non-destructive testing on concrete. However, it is a very challenging task to estimate the correct value of the strength of concrete or cement as it is depending on various factors. The present research work is focussed on the impact of zinc oxide (ZnO) nano-particles on the compressive strength of the cement mortar. To investigate the modifed compressive strength of the mortar incorporated with ZnO nano-particles, four diferent types of mixes were prepared with 0%, 0.25%, 0.5%, and 0.75% of the ZnO nanoparticle by the weight cement, respectively. Experimental results show the enhancement in compressive strength up to 0.5%, later on, strength is slightly decreased. By considering the experimental results of cement strength, three diferent models are proposed to predict the strength of cement mortar as analysis of covariance (ANCOVA), neural network (NN), and principal component regression (PCR). These models also validate the results of experimentation by showing the optimum results at 0.5% of the addition of ZnO nano-particles. These models are trained and tested in excel programming for thirty-six standard cement specimens. At the end of the work, each model is compared with others. Out of three models, the NN model can predict the reliable results for the compressive strength. However, the PCR model is in second place after the NN model though its value of  $R^2$  is lesser than the ANCOVA model. PCR gives less residue as compared to ANCOVA. For the prediction of the strength of mortar, ANCOVA is not so signifcant as compared to the other two models due to the residuals of ANCOVA models are the largest value, though its  $R^2$  value is more than the PCR model.

**Keywords** Compressive strength · Nano-particles · Analysis of covariance · Neural network model · Principal component regression

# **Introduction**

Cement is one of the most often used materials by humans, and the compressive strength of structural concrete is determined by the concrete's numerous constituents. It is also one of the most important components that provide concrete its strength. The strength of the cement determines the concrete's performance. Standard specimens were created and cured at various days for the curing in the laboratory

 $\boxtimes$  Hiteshkumar Patil hitusp@gmail.com Arunkumar Dwivedi akd321@gmail.com

School of Engineering and Technology, Sandip University, Nasik, MS 422002, India

to test the strength of cement. Some concrete factors, such as cement, aggregates, water-cement ratio, curing period, have an impact on the strength of the concrete. The various regression [\[1](#page-13-0), [2](#page-13-1)] approaches are used to calculate the strength of cement using known procedures. The trend of evaluating cement strength using models that are only based on data has been expanding over the last few years. Many researchers are currently considering various approaches to estimate cement and concrete strength, such as artifcial neural networks (ANN)) [[3–](#page-13-2)[5\]](#page-13-3), principal component regression  $[6–8]$  $[6–8]$  $[6–8]$  $[6–8]$ , fuzzy logic  $[9, 10]$  $[9, 10]$  $[9, 10]$  $[9, 10]$  $[9, 10]$ , and machine learning methods [[11\]](#page-13-8).

Many researchers have looked at estimating the particular properties of concrete. Using design of experiment analysis, Salem Alsanusi and Loubna Bentaher evaluated the compressive strength of concrete [\[12](#page-13-9)]. Moutassem et al. [[13](#page-14-0)] anticipated theoretical and phenomenological models for predicting concrete qualities. ANN is also used to

<sup>&</sup>lt;sup>1</sup> KBC North Maharashtra University, Umavi Nagar, Jalgaon, MS 425001, India

investigate displacement in a concrete reinforced building [\[14](#page-14-1)]. Joseph Mwati Marangu researched the use of machine learning (support vector machine) to estimate the strength of concrete [[15\]](#page-14-2).

Hamid Eskandari-Naddaf et al. [\[16](#page-14-3)] used an artifcial neural network (ANN) to forecast the compressive strength of mortar mixtures with various cement strength classes. With improvements in cement class, ANN exhibits an improvement in mortar compressive strength. Because cement is made up of random, complicated, multi-scale composites, predicting its elastic characteristics is difficult. Finite element approaches, in combination with knowledge of individual phase modulus and a cement paste microstructure development model, are utilised by Haecker [[17](#page-14-4)] to quantitatively predict elastic modulus as a function of degree of hydration as determined by loss on ignition. For degrees of hydration of 50% or greater, and for a range of water cement ratios, comparisons between model predictions and experimental results are good. Kavita Verma et al. [[18\]](#page-14-5) offer a fnite element model for cement strength prediction. They also utilise the ANN to forecast the strength of cement mortar in various sorts of environmental conditions, confrming that the ANN model's prediction is the best. The amount of sodium chloride, chemical admixtures, and cement grade all have an impact on the compressive strength of cement mortar. As a result, Hamid Eskandari et al. [\[19](#page-14-6)] devised an artifcial neural network model to estimate the compressive strength of mortar for various cement grades and sodium chloride (NaCl) percentages. Additionally, Shaqadan et al. [\[20\]](#page-14-7) proposed the Relevance Vector Machine (RVM) and Random Forest (RF) models, and the study found that RVM predicts cement mortar strength better than RF. The ANN, correct orthogonal decomposition, and regression analysis models were compared to the created mathematical model by Kisan Bidkar et al. [[21](#page-14-8)]. Akbar Ghanbari et al. [\[22\]](#page-14-9) employ micromechanical parametric models to estimate the plastic viscosity of self-compacting steel fbre reinforced concrete (SCFRC) based on the observed plastic viscosity of the mixture. The study of Naresh Kumar Nagwani et al. [\[23\]](#page-14-10) utilizes the cluster regression approach to estimate the concrete's compressive strength. Clustering and regression together will guarantee that the dependent and independent variables' curves are more accurately ftted. Huaicheng Chen et al. [[24\]](#page-14-11) deploy a support vector machine (SVM) model to estimate the compressive strength of mortars subjected to sulphate assault. The possibility of utilising artifcial neural networks (ANNs) modelling to forecast the characteristics of self-compacting concrete (SCC) incorporating fy ash as a cement substitute is investigated by Omar Belalia Douma et al. [\[25](#page-14-12)].

The current investigation is focussed on the estimation and comparision of the strength of cement mortar incorporated with nano ZnO particles by using diferent types of models such as analysis of covariance (ANCOVA), artifcial neural network (ANN), and principal component regression (PCR). The outline of the research work is shown in Fig. [1](#page-2-0)c.

## **Materials and methods**

#### **Materials**

*Cement*: Pozzolana Portland Cement confirming to IS 1489:1991  $[26]$  $[26]$  is used in this investigation. Table [1\(](#page-3-0)a) and Table [1\(](#page-3-0)b) show the chemical and physical properties of cement, respectively.

*Fine aggregate:* Locally available fne aggregate in the river is used for the experimentation, it is confrming to IS 383: 1970 [[27\]](#page-14-14) with Zone II utilizes for the experimentation. The properties of cement and fne aggregate shown in Table [1\(](#page-3-0)c).

*Zinc oxide (ZnO) nano-particles:* Zinc ash or zinc oxide is a by-product of the coating industry, which occurs as a fne powder and grey in colour. Figure [1a](#page-2-0) shows an average particle size of  $ZnO$  is 50 nm and a density of 263 kg/cm<sup>3</sup>. The Chemical composition of the ZnO nano-particles is shown in Table  $1(d)$  $1(d)$ .

*Characterisation of Zno particles*: XRD fgure of ZnO nanoparticles given in Fig. [1b](#page-2-0). The XRD peaks show the materials in the nano-scale series. From these peak intensity, width and position, full width at half maximum data (FWHM) can be determine. The peaks situated at 30.88°, 33.539°, 35.364°, 46.659°, 55.718°, 61.988°, 67.076°, 68.213°.

 $D = (0.9 \times \lambda)/(\beta \times \cos \theta)$  Scherrer equation [28],

where  $\lambda$  = wavelength,  $\beta$  = full width at half maximum (FWHM)of difraction peak. The average practical size is 48.69 nm at an angle 35.36°, it is calculated as Scherrer Equation. The widening of XRD peaks shows the presence of nanoscale particles in the materials. XRD pattern analysis may be used to identify peak intensity, location, and breadth-full width at half maximum data. SEM pictures demonstrate the formation of ZnO nano-particles. SEM confrms the approximately spherical form, and most particles have some faceting. The selected region electron difraction pattern confrms nano-crystals preferred orientation over conventional crystals [\[29](#page-14-15), [30](#page-14-16)]. SEM images, which reveal the formation of ZnO particles, also confrm the hexagonal plane.



<span id="page-2-0"></span>**Fig. 1 a** SEM image of ZnO **b** X-RD results of ZnO **c** Outline of the research work **d** Nano ZnO

# **Methods**

Experiments start with the preparation of 4 types of mixes. In these mixes, the amount of addition of ZnO nano-particles varies from 0%, 0.25%, 0.5% and 0.75% by weight of the cement. The mix proportions and its details are shown in Table [2](#page-3-1). The compressive strength of cement mortar was checked as per IS 4031 (part  $6$ ) [\[31](#page-14-17)].

The outline of the research work is shown in Fig. [1](#page-2-0)c. After testing the specimens of all mixes, three types of models were prepared for the prediction of the strength of cement. Table  $3(a)$  shows the input parameters for the various models and Table [3\(](#page-4-0)b) shows the characteristics of specimens after casting. At the end of the work, results of all models were compared with each other and fnd out the best model ft for further experimentation. The predicted values from models and experimental values were calculated. The residuals of each model were again compared and based on this, the best-ftted model was customized for this work.

<span id="page-3-0"></span>**Table 1** (a) Chemical and physical properties of the cement (b) properties of fne aggregates (c) chemical composistion of zinc oxide (%)

Cement			Notation			% Mass
(a) Chemical composition of cement						
Calcium oxide			Cao(C)			65%
Silicon dioxide		SiO <sub>2</sub> (S)			21%	
Aluminium oxide			$\text{Al}_2\text{O}_3(\text{A})$			5%
Ferric oxide			Fe <sub>2</sub> O <sub>3</sub> (F)			$2\%$
Sulfer trioxide			$SO_3(S)$			2
(b) Physical properties of cement						
Specific gravity:						3.15
Fineness $(\%)$ :						0.89
Consistency $(\%)$ :						32
Initial setting TIME (IST) Min:						143
Final setting time (FST) Min:						205
Soundness (mm):						0.6
(c) Properties of fine aggregates						
Fineness modulus (%)						3.46
Specific gravity						2.71
Bulk density $(kg/m3)$						1549
Water absorption $(\%)$						0.22
(d) Chemical composistion of zinc oxide $(\%)$						
Fe Zinc	Pb	Cd	Cu	Mg	Sn	AI
63 0.66	0.24	0.0011	0.08	0.02	0.12	1.62

<span id="page-3-1"></span>**Table 2** Mix proportions



## **Models**

## **Analysis of covariance (ANCOVA)**

ANCOVA is used for the assessment of the main efect of categorical variables on the main (dependent) variables. It is also used for limiting the efect of continuous variables on the dependent variables. ANCOVA is blended with linear regression (LR) and analysis of variance (ANOVA) as the same type of dependent variables is there, so the model alters linearly and the hypothesis becomes identical. If p is the number of quantitative variables, and q the number of factors (the qualitative variables including the interactions between qualitative variables), the ANCOVA model is written as [[32](#page-14-18)]:

$$
y_i = \beta_0 + \sum_{j=1}^p \beta_j x_{ij} + \sum_{j=1}^q \beta_{k(i,j),j} + \varepsilon_i
$$
 (1)

where  $y_i$  is the value observed for the dependent variable for observation *i*, *xij* is the value taken by quantitative variable *j* for observation *i*, *k*(*i*,*j*) is the index of the category of factor *j* for observation *i* and  $\varepsilon$ <sub>*i*</sub> is the error of the model. By ANCOVA, here we wanted to determine the compressive strength. Use of this model to predict compressive strength as the dependent variable and zinc ash, cement to zinc ash ratio, water to cement ratio as quantitative variables, and curing days as qualitative variables as the portion of multiple linear models.

Compressive strength=Function (zinc ash, cement to zinc ash ratio, water to cement ratio and curing days).

## **Neural network (NN)**

Due to the strength, the user-friendly approach and the elasticity of Neural Network model is widely preferred model for the prediction. This is most useful under the multifaced circumstances. Radial basis function (RBF) [[33,](#page-14-19) [34\]](#page-14-20) and multilayer perceptron (MLP) [[35,](#page-14-21) [36\]](#page-14-22) applications are used

<span id="page-4-0"></span>**Table 3** Input details



Where  $X1 = ZA$  (gm);  $X2 = C/ZA$ ;  $X3 = W/C$ ;  $X4 =$ Curing Days



<span id="page-5-0"></span>**Fig. 2** General architecture of neural networks

to predict. The target values are compared with predicted values and this task is supervised by RBF & MLP models.

Figure [2](#page-5-0) describes the architecture of neural network. In this frst layer is input layers which include the predictors. The second layer (hidden layer) includes the units or ignored nodes. The value of each node is the function of the predictors, MLP, and RBF extracted required function value as per their requirement. The third (output) layer includes the reply or output of the model. MLP involved a second hidden layer in which each node is the function of frst layer nodes. In this work zinc ash, cement to zinc ash ratio, water to cement ratio & curing days are the input parameters and compressive strength is the output parameter.

#### **Principal component regression (PCR)**

Generally, the Principal component Regression technique is adopted when there is a multicollinearity problem is arising in data for the linear regression. Multicollinearity causes estimation of least squares neutral values with big variance, so its values are so far from its true values and create more standard errors. In such a case, PCR reduces the standard values and gives accurate prediction values. PCR [\[37](#page-14-23)] is divided into three parts. In first step, principal component factors are extracted from the variables and in second step ordinary least square regression is performed between dependent parameters and the component which were extracted from the frst step. The following Eq. [\(2](#page-5-1)) is the basic regression equation for the PCR model [[38\]](#page-14-24).

$$
Y = BX + e \tag{2}
$$

where  $Y =$ Dependent variables,  $X =$ Independent Variables, *B*=regression coefficient and  $e$ =error or residuals. In this work, Zinc Ash, Cement to zinc ash ratio, water to cement ratio and curing days are the input parameters and compressive strength is the output parameter.

## **Results and discussion**

In this section, the experimental results are compared with the ANCOVA, NN, and PCR models prediction. These results showed that ZnO nano-particles enhance the compressive strength of the cement mortar signifcantly, the detailed discussion about the result is given below.

## **ANCOVA model**

In Table [4,](#page-6-0) output parameters of the ANCOVA model are given. Using the Backward variables selection method, 4 variables have been retained in the model. Given the  $R^2$ , 99.40% of the variability of the dependent variable Strength (Mpa) is explained by the 4 explanatory variables. Given the *p* value of the *F* statistic computed in the ANOVA table, and given the signifcant level of 5%, the information brought by the explanatory variables is signifcantly better than what a basic mean would bring. Form Table [4\(](#page-6-0)a), the ANCOVA model  $R^2$  value is 0.9994. It means all variables correlated and signifcant for the prediction of the strength. As this value is closer to 1, the model is the best-ftted model. From the analysis of variance in Table  $4(b)$  $4(b)$  *F* value <0.0001, it shows that all independent variables in the model are signifcant. Abhijit Chatterjee et al. [\[32](#page-14-18)] and Okan et al. [[39\]](#page-14-25) were also reported similar types of results.

Figure [3](#page-7-0) shows the results of ANCOVA models in the graphical format. Figure [3](#page-7-0)a gives the value of  $R^2$  = 0.9994 of predicted value and the observed value of the ANCOVA model. Figure [3b](#page-7-0) and Fig. [3](#page-7-0)c give the standard residue of the training and validation set, the value of the standard error is between−1.4 and 2.1 for active data and−2 to 2 for validation data for the ANCOVA.

## **Neural network model**

<span id="page-5-1"></span>For research work, 36 specimens were cast. Out of 36 the 28 specimens (77.8%) were considered as training specimens and 8 specimens (22.2%) were considered as testing purposes. The summary of the model processing is given in Table  $5(a)$  $5(a)$  and Table  $5(b)$  shows the network information for the model. Results of model summary are displayed in Table  $5(c)$  and the importance of the variables shows in Table [5](#page-8-0)(d). The architecture of the neural network model is shown in Fig. [4a](#page-9-0). Figure [4](#page-9-0)b shows the plot of predicted vs observed values in this value for  $R^2$  is 0.999 which shows the model best ftted. Figure [4c](#page-9-0) shows the residuals of the NN model which has a range between−0.7 to 1. The obtained results in research work are similar to khademi et al. [\[9](#page-13-6)] and Sudarshan et al. [[4\]](#page-13-10). As residues are less, so it is suggested that the model predicts the accurate values. This model also

<span id="page-6-0"></span>

gives normalizes importance to each variable. The validation of the neural network models is its residuals as from Fig. [4](#page-9-0)b and c is very minimum means the prediction of the models is very accurate.

## **Principal component regression model**

Figure [5a](#page-10-0) shows the scree plot for the PCR model. It shows that the model describes 50% of the variability, so it is possible to present variable on the one axis. Table [6](#page-11-0) provides the information of output for the model of PCR. From Table  $6(a)$ and Fig. [5](#page-10-0)b the goodness of the fit statistics, the value of  $R^2$ is 0. 944, so that the PCR model is best ftted. The values of the standard residue of active and validation data are shown in Fig. [5b](#page-10-0) and c and the range of residue for active data is between−1.17 and 1.8 and validation data is−1.2 to 1.6.

So, the prediction of the PCR models also gives accurate values. The same types of results also observed by Islam et al. [[6](#page-13-4)] in their work. PCR models provide the equation given in Table  $6(d)$  $6(d)$  for the prediction of the compressive strength of mortar, this equation represents the 93.2% of the variability.

Table [7](#page-12-0)(a) shows the overall comparison between compressive strength by actual experimental testing and compressive strength predicted by diferent models. Table [7](#page-12-0)(a) and Fig. [6a](#page-13-11) display the compressive strength of all mixers enhances with ZnO nano-particles addition continue up to 0.5% in cement, later on, the strength of cement mortar is decreased. Table [7\(](#page-12-0)b) shows the comparison of all models, in that  $R<sup>2</sup>$  and minimum and maximum residues are compared, the values of  $R^2$  are very closer to 1 for each model but from this, it is not clear that which model is best ftted but with



<span id="page-7-0"></span>**Fig. 3** Graphical results of ANCOVA models

the help of residuals plot given in Fig. [6](#page-13-11)b, it is possible to suggest the best model for the prediction of the compressive strength of the mortar.

Out of three models, the Neural Network produces the lesser value of the residue that means it is the best model for the prediction of the strength of cement mortar. It has minimum residue values as−0.7 and 1. Figure [6b](#page-13-11) shows the comparison of overall performance by the models. This indicates that all models give the optimum response at 0.5% of addition afterwards it shows declination in the results of the compressive strength of the mortar. This is directly validated the outcomes of the research. The decreases in the strength of mortar may be causes due to ZnO nano-particles formed the crystalline layer of  $CaZn_2(OH)_2·2H_2O$  around the cement

## <span id="page-8-0"></span>**Table 5** Output for NN model







<span id="page-9-0"></span>**Fig. 4** Graphical outputs of the NN model



<span id="page-10-0"></span>**Fig. 5** Graphical representation of output of PCR model

particles. As per the available literature, some of reported that the analysis of X-ray difraction (XRD) of calcium zinc hydrate is formed with ZnO nano-particles which restrict the hydration reaction and result in the formation of fewer hydrates and ultimately it decreases the compressive strength of the mortar. The similar types of results were reported by Riahi et al. [[40\]](#page-14-26) Kantharia et al. [[41\]](#page-14-27) and Arefi et al. [[42](#page-14-28)].

# **Conclusions**

The outcomes of the experimentation show that mortars containing ZnO nano-particles have expressively higher strength as compared with mortar without the ZnO. The optimum % of the addition of ZnO nano-particles is 0.5%. To validate and prediction of the experimental results, three diferent mathematical modelings reported. These three models based on Analysis of variance, Neural Network, and Principal Component Regression. Thirty-six standard specimens of cement paste with nanomaterials were casted. The compressive strength is considered as a function of the zinc oxide nano-particles, cement to zinc oxide ratio, water to cement ratio, and curing days. It is concluded that the neural network (NN) model is the best ftted and more accurate model than other models. Principal component regression (PCR) predicts an accurate value of the compressive strength as compared to the analysis of variance (ANCOVA) model. Although the  $R^2$  value of the ANCOVA model is better than the PCR model, the residuals of the ANCOVA model are more than the PCR model. This shows that PCR gives accurate and better prediction than ANCOVA. In all NN and PCR models can be confdently customized to the estimation of the compressive strength of cement mortar instead of vast and costly methods. The results of NN and PCR models also helpful for material selection and its mixed design process.

## <span id="page-11-0"></span>**Table 6** Outputs of PCR model



(d) *Equation of the model*

Strength= −104.47–18.90×X1-4.1E-03×X2+442.93×X3+1.233×X4

<span id="page-12-0"></span>





<span id="page-13-11"></span>**Fig. 6** Comparison of overall performance of the mixes and models

## **Declarations**

**Conflict of interest** On behalf of all authors, the corresponding author states that there is no confict of interest.

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