NIPES

Journal of Materials Engineering, Structures and Computation

Journal homepage: www.nipesjournals.org.ng

Journal of Materials Engineering, Structures and Computation

Modelling the Effect of High Temperatures on Concrete Made with Discarded Bottle Powder (DBP) and Metakaolin (MTK)

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ARTICLE INFORMATION

Article history:
Received 05 May 2023
Revised 24 May 2023
Accepted 26 May 2023
Available online 12 June 2023

Keywords:
Compressive Strength, Flexural Strength, High Temperatures, Metakaolin, (MTK) Discarded Bottle Powder (DBP) and Modelling.

https://doi.org/10.5281/zenodo.8027759

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ABSTRACT

This study models the effect of high temperatures of 100°C, 200°C, 300°C, 400°C and 500°C on blended concretes made with discarded bottle powder (DBP) and metakaolin (MTK). The blended concrete of two mixes containing ten (10%) of discarded bottle powder only and blended concretes of three mixes containing ten (10%) of discarded bottle powder with 5%, 10%, 15% and 20% metakaolin were produced. Six different mixes were produced for the concrete cubes and beams. Seventy-two (72) concrete cubes of sizes 150mm x 150mm x 150mm were produced to determine the compressive strengths ***(****before and after heating the hardened samples****)*** *and Seventy-two (72) concrete beams of sizes 150mm x150mm x 500mm were also produced to determine the flexural strengths* ***(****before and after heating the hardened samples****)****. Slump test was conducted on each of the six mixes produced to determine its workability. The concretes were cured for 28 days by complete immersion in water. The concrete cubes and beams were heated at the above-mentioned temperatures before testing using Electrical Motorized compression Machine and Universal Testing Machine respectively. The results showed that the strengths generally decreased as the percentage of metakaolin (MTK) increased****,*** *with* ***i****ncreased temperature. Concrete cubes made with 5% MTK achieved the 28days target strength of 25N/mm^2 at temperature of 100°C. The optimum replacement level is MTK 5% DBP 10% at100°C. The data obtained were analyzed using response surface regression and Analyses of Variance (ANOVA) in the Minitab 16 Statistical Software. Models were developed to predict the compressive and tensile strengths with percentage mix and temperature as independent predictor variables. The t-Test indicates that there is no statistical significant difference between the experimental and predicted strength value at 0.05 level of significant. The correlation coefficients, R^2 of 96.57% and 91.89% for compressive and flexural strengths model, indicate that the model prediction gave a good correlation factor which implies that the model predictions are accurate.*

1. Introduction

Recent change in research is tilted towards developing cheap, available construction materials which is necessitated by the cost of cement and the need to maintain environmental and ecological balance in the ecosystem. The construction industry today is considered the largest consumer of natural resources such as coarse and fine aggregates, timber, ceramics, Pvc, Pipes, etc. [1, 2] Research has revealed that over ten billion tonnes of concrete are produced worldwide and used annually [3]. The extensive use of concrete as a structural material has led to the need to fully understand the resistance of concrete at high temperature. However, there are some physical and chemical transformations that occur when concrete is subjected to increased temperature, such as spalling and cracks which at times might lead to total collapse of the entire structure. Concrete characteristics such as color, compressive strength, elasticity, density and surface appearance are mostly affected by high temperature [1, 2, 4, 5]. [6] is of the opinion that cement replacement with pozzolanic material such as metakaolin (MTK) is one of the most efficient methods of improving the fire resistance of concrete. Fire resistance of concrete is highly dependent on many factors such as constituent materials, particularly the pozzolanic materials. However, strength reductions have been reported in many literatures due to the high temperature, test condition, varieties of constituent materials used for concrete production [7]. [4] reported that the behavior of concrete subjected to high temperature depend on rate of heating, peak temperature, phase transformation and thermal incompatibility between cement paste and aggregates. Combined use of DBP and MTK mineral admixture can lead to economic advantages and technological improvements [8]. One of the advantages offered by SCMs blended concrete such as rice husk ash, waste glass powder, fly ash, and metakaolin e.t.c. Ordinary Portland cement (OPC) is one of the binders mostly used in building construction and concrete products. In 2012, global cement production was estimated to be about 3.6 billion tonnes while the green gas carbon dioxide (CO_2) associated with the cement production in the same year was estimated to be 2 million tonnes [9]. [10, 11, 12, 13, 14, 15, 14, 16, 17] have shown that the use of supplementary cementation materials (SCMs) is environmentally-friendly and less in cost when compared to ordinary Portland cement. In addition to improving concrete properties, it also provides solution to the depletion of the natural resources thereby helping in maintaining the natural environment. The risk of fire outbreak increases with modernization, and this is because modern surroundings are full of objects made from highly flammable materials, which are potential ignition sources [4]. All over the world, concrete structures are exposed to high risk of fire and hazards daily, resulting in human and material losses. Human safety in the case of fire is one of the major considerations in co-operated in the design of buildings. It is extremely necessary to have a complete knowledge about the behavior of all construction materials at increased temperature before using them as structural elements. In Nigeria between October 2008 and April 2009, there have been reported cases of about fifty one fire outbreaks in Lagos State alone, with almost non-recovery of the whole construction material including concrete [18, 19]. High temperature has negative effects on concrete and increases irrecoverable deformation [20, 21]. [22, 23] reported that the fire resistance of concrete can be improved by partial replacement of cement with pozzolanic materials such as rice husk ash, fly ash, and sawdust ash, metakaolin (MTK), discarded bottle powder (DBP) and so on.

Use of discarded bottle powder not only help in reducing the cost of cement and concrete manufacturing, but also has numerous other benefits such as reduction in landfill cost, saving in energy, and protecting the environment from possible pollution effects. Discarded bottle is readily available in most part of the world. However, according to the United Nation, the estimation of solid waste in 2021 is about 8.3 billion tonnes, out of which 12% is waste from glass. The non-biodegradable nature of discarded bottle makes it disposal to landfills a problem, while cement and concrete industries can provide an environmentally friendly means of disposing it. Metakaolin (MTK) is obtained by burning kaolin between temperature ranges of 600-900°C

[9]. Metakaolin improves strength and concrete durability through the acceleration of ordinary Portland cement (OPC) hydration and the pozzolanic reaction with calcium hydroxide
$Ca(OH)_2$. It had proven to have good fire resistance when blended with cement in concrete up to 400°C [4, 24]]. Therefore, this study will assess the influence of metakaolin on the strength of concrete containing discarded bottle powder at high temperature and the results obtained were used

to develop statistical models for predicting compressive and flexural strengths of discarded bottle powder concrete (DBPC) and metakaolin concrete (MTKC).

2. Materials and Methods

2.1 Materials

The discarded bottles were collected from glass dealers at Okwei Street in Onitsha Metropolis, Anambra State. The discarded bottles were sorted out, washed and sun dried before crushing using mechanical crusher to smallest possible sizes. It was sieved using 75µm British Standard sieve at the Structures Laboratory of Hartland Construction Company, Umudike, Abia State. Kaolin was purchased from paint manufacturers in Onitsha, Anambra State and the dried kaolin was calcined by burning at temperature range of 600°C to 900°C as recommended by (Rashad, 2013) to produce metakaolin. Ordinary Portland cement (Dangote brand) used for this practical work and the properties of the cement conform to ES 197 (1992) specifications. The coarse aggregate used was normal weight aggregates from indigenous rock, obtained from Hartland Construction site, Umudike, Abia State and the fine aggregates used was also obtained from the same Construction Site. The water used for the study was tap water obtained from tap source free from impurities.

2.2 Features of Discarded Bottle Powder (DBP) and Metakaolin (MTK)

The chemical analyses of the samples (Discarded bottle powder and metakaolin) were conducted at the Alpha Research Chemical Laboratory, Awka, Anambra State. The tests were conducted in accordance with ASTM C 311 (2011) specifications. The tests were carried out to determine the oxides composition of the representative samples (Discarded bottle Powder, Metakaolin and cement. The results are shown in Table 1

2.3 Production of Concrete Using Constituent Materials

Six (6) different mixes were produced. Discarded bottle powder (DBP) and metakaolin (MTK) were used as cement replacement in concrete production. The level of replacements were 0%, 5%, 10%, 15%, 20% for metakaolin (MTK) while the discarded bottle powder (DBP) was kept constant at 10% cement replacement in line with certain recommendations (Schwart, et al., 2008; Matos and Sousa, 2012; Khimiri, et al., 2013; and Shi, et al., 2005). For each of the mix, the test was repeated twice and the average of the two results was recorded and used. Table 1 shows the quantity of the constituent materials used for the production of concrete cubes and beams in line with American Concrete Institute (ACI) 211 method.

Table 1. Mix Proportion of Constituent Materials used for the concrete production

% Mix	Constituents Materials (kg/m^3)					
	Binders			Aggregates		
	DBP (kg/m^3)	MTK (kg /m^3)	Cement (kg/m^3)	C.Agg. (kg/m^3)	F.Agg. (kg/m^3)	Water (kg/m^3)
MTK0% DBP0% (Control)	0	0	385	1296	558	185
MTK0% DBP10%	32	0	346	1296	558	185
MTK5% DBP10%	32	16	329	1296	558	185

MTK10% DBP10%	32	33	311	1296	558	185
MTK15% DBP10%	32	49	294	1296	558	185
MTK20% DBP10%	32	65	277	1296	558	185

2.4 Production of Concrete Cubes and Beams

Concrete cubes of size 150mm x 150mm x 150mm were produced to determine the compressive strength before and after heating the hardened samples. Concrete beams of size 100mm x 100mm x 500mm were also produced from the same mix to determine the flexural strength before and after heating the hardened concretes.

2.5 Slump Test

Slump test was conducted on each of the six (6) mixes produced to determine its workability. The test was conducted at Hartland Construction Company located in Umudike, Abia State. The test was conducted in accordance with BS EN 12350: part 2 (1999) specifications.

2.6 Curing of Concrete Cubes and Beams

The concrete cubes and beams were cured for a maximum of 28 days by complete immersion in water. Curing of concrete cubes and beams were performed in accordance with BS EN 12390, part 2 (2000) specifications.

2.7 Heating of Concrete Cubes and Beams

The cured concrete cubes and beams were heated at 100°C, 200°C, 300°C, 400°C and 500°C. The temperature was maintained for a period of 30 minutes to achieve the thermal steady state. The heating was conducted at the Structural Laboratory of Hartland Construction Company, Umudike, Abia State. The heating was carried out in accordance with BS 8110 part 1 (1997) specifications.

2.8 Compressive Strength Test

The concrete cubes were prepared in accordance with BS EN 12390, part 3: (2002). It was carried out on hardened concrete cubes of 150mm x 150mm x 150mm. The samples were tested using ELE Motorized Compression Machine in accordance with BS EN 12390, part 4: (2000) specifications at the Structural Laboratory of Hartland Construction Company, Umudike, Abia State. The compressive strengths of the concrete cubes were determined using Equation 1

$$\text{Compressive Strength} = \text{Failure Load} / \text{Area of Specimens} = P/A \qquad (1)$$

Where failure load is measured in Kilo Newton KN and area of the specimen is measured in millimeter square (mm^2).

2.9 Flexural Strength Test

Flexural strength test was carried out on the hardened concrete beams of 100mm x 100mm x 500mm. The samples were tested using Universal Testing Machine for three points loading. The flexural strength of the concrete beams was determined at these temperatures-100°C, 200°C, 300°C, 400°C, 500°C. The test was conducted at the Structures Laboratory of Hartland Construction Company, Umudike, Abia State, in accordance with BS EN 12390, part 5 (2000) specifications. The flexural strength is expressed as a modulus of rupture (MOR) measured in Newton per millimeter square (N/mm^2) and was determined from Equation 2.

$$\text{Modulus of rupture (MOR)} = PL/bd^2 \qquad (2)$$

Where P = Maximum Load measured in Kilo Newton (kN)

L = Span of the beam = 500mm
d = Depth of the beam = 100mm
b = Breadth of the beam = 100mm

3 Results and Discussion

3.1 Oxide Composition and Physical Properties of Metakaolin (MTK)

Table 2. Oxide Composition of Metakaolin (MTK)

Parameter	Silicon oxide (SiO_2)	Aluminium oxide (Al_2O_3)	Iron oxide (Fe_2O_3)	Calcium oxide (CaO)	Magnesium (oxide) MgO
Oxide (%)	53.00	37.00	2.50	1.60	0.14

Table 3. Physical Properties of Metakaolin (MTK)

Parameter	Specific gravity	Loss on ignition (LOI)	pH
Value	2.98	0.08	9.50

Table 2 shows the test results for the oxide analyses conducted on MTK. The result indicates the presence of similar oxides to those of cement and other pozzolanas. The sum of the oxides of silicon, and aluminum was 92.5% which exceeds the 70% minimum specified by ASTMC 618 (2012) for raw or calcined pozzolana (class N). The presence of these oxides determine the amount of C_3S, C_2S and C_3A, hence the performance of the MTK blended concrete. These compounds contribute to the early and later strength as well as the setting characteristics of concrete (Neville and Brooks, 1997). The silica content of the ash is another important factor because when ash from certain agricultural by products like rice husk and bagasse is added to cement, the silica reacts with Ca $(OH)_2$ to form additional C-S-H in the hydrated cement matrix which increases the density of the matrix and refines the pore structure. Table 3 shows the physical properties of metakaolin (MTK.) The loss on ignition (LOI) is a measure of the extent of carbonation and hydration of free lime and free magnesia due to atmospheric exposure. The LOI of MTK determined was 0.08%. This value falls below the maximum of 10% specified by ASTMC 618 (2012). The low LOI of MTK is an indication of the presence of very small amount of impurities, these impurities are mainly carbon which may increase the water demand of the concrete (Kevern and Wang, 2012). The specific gravity of MTK was 2.98, while that of Dangote cement was 3.15. This indicates that MTK is lighter than cement and more volume of MTK will be needed to replace equal weight cement in concrete. The pH of MTK determined was 9.5. This value indicates that MTK is neither acidic ($pH < 7.0$) nor alkaline ($pH > 11$) but neutral (pH of between 7-9).

Table 4 shows the test results for the oxide analyses conducted on discarded bottle powder. The result shows the presence of similar oxides to those of cement and other pozzolanas. The sum of the oxides of silicon, iron and aluminum is 71% which exceeds the 70% minimum specified by ASTMC 618 (2012) for raw calcined pozzolana (class N).

3.2 Oxide Composition and Physical Properties of Discarded Bottle Powder

Table 4. Oxide Composition of Discarded Bottle Powder (DBP)

Parameter	**Silicon oxide (SiO$_2$)**	**Aluminum oxide (Al$_2$O$_3$)**	**Iron oxide (Fe$_2$ O$_3$)**	**Calcium oxide (CaO)**	**Magnesium oxide (MgO)**
Oxide (%)	65.00	3.90	2.11	10.80	0.1

Table 5. Physical properties of Discarded Bottle Powder (DBP)

Parameter	**Specific Gravity**	**Loss on ignition (LOI)**	**pH**
Value	2.60	0.06	9.80

The presence of these oxides determine the amount of C_3 S, C_2S and C_3A hence the performance of the DBP in the blended concrete. These compounds contribute to the early and later strength as well as the setting characteristics of concrete (Neville and Brooks, 1997). The silica content of DBP is another important factor because when added to cement, the silica reacts with Ca $(OH)_2$ to form additional C-S-H in the hydrated cement matrix which increases the density of the matrix and refines the pore structure. Table 5 shows the physical properties of discarded bottle powder (DBP). The loss on ignition (LOI) is a measure of the extent of carbonation and hydration of free lime and free magnesia due to atmospheric exposure. The LOI of DBP determined was 0.06% and the value falls below the maximum of 10% specified by ASTMC 618 (2012). The low LOI (0.06%) of DBP indicates that there was very small amount of impurities which are mainly carbon and may increase the water demand of the concrete containing DBP and consequently affect the effectiveness of incorporating DBP in concrete (Kevern and Wang, 2012). The specific gravity of DBP determined was 2.60, while that of cement was 3.15. This indicates that DBP is lighter than cement and more volume of DBP will be required to replace equal weight of cement in concrete. The pH of DBP was determined to be 9.80, this value shows that the DBP is neither acid (pH< 7.0) nor alkaline (pH >11) but neutral (pH between 7 - 9).

3.3 Slump Test Results of the Blended Concrete

Table 6. Slump Test Result of the blended Concrete

Percentage Mix (%)	**Slump (mm)**
MTK0 DBP0 (control)	30
MTK0 DBPI0	25
MTK5 DBP10	20
MTK10 DBP10	15
MTK 15 DBP 10	10
MTK 20 DBP 10	8

The results of the slump test carried out on concrete with varying percentages of MTK as cement replacement are presented in Table 6. Workability is a vital property of concrete because it determines the amount of work required for placing and compacting concrete. Concrete slump can be classified as true slump (up to 125mm), shear slump (up to 150mm) and collapse slump (150-250mm). All the experimental slump values obtained falls within the category of true slump type and suitable for concrete works. The result shows that the slump decreases with increase in the amount of MTK which indicates that more water is required to maintain the same consistency as the metakaolin (MTK) increases. Concrete containing MTK of 5%, 10% and 15% with slump values of 20mm, 15mm and 10mm respectively falls within the limit of class S1 (10mm – 40mm) specified by BS 12350 (1999) and approved for concrete works. Due to the adverse effect of metakaolin (MTK) on concrete workability, cement replacement of not more than MTK 10% DBP 10% may be considered. Consequently, mechanical or hand vibration may be applicable when metakaolin (MTK) is intended for use as pozzolana in concrete.

3.4 Compressive Strength Test Results

Table 7 Compressive Strength Test Results obtained for the blended concrete at Different temp.

% Mix	25^0C	100^0C	200^0C	300^0C	400^0C	500^0C
MTK0 DBP 0	35.75	34.48	33.62	30.13	26.80	23.05
MTK0 DBP 10	28.87	27.46	27.81	25.78	26.12	22.54
MTK5 DBP 10	24.64	24.47	23.05	21.92	21.94	21.20
MTK10 DBP10	23.34	22.60	21.52	20.66	18.51	16.51
MTK15 DBP 10	23.12	21.34	22.17	19.17	17.86	14.82
MTK20 DBP10	22.67	20.95	20.54	18.25	17.72	14.55

Figure 1 shows the plot of compressive strength of OPC-DBP-MTK concrete versus percentages of metakaolin (MTK) used to replace cement. It can be seen that the strength decreased as the percentage of MTK increased. At temperatures of 25^0C to 500^0C, the strength decreased directly with increase in MTK content. This could be attributed to the replacement of cement with MTK in concrete which resulted in the reduction of tri-calcium silicates (C_3S), the main strength contributing compound, the loss of moisture which prevent long term hydration and the destruction of an active strength generating ingredients like cement and aggregate due to continuous rise in temperature (Chandam, *et al.*, 2013).

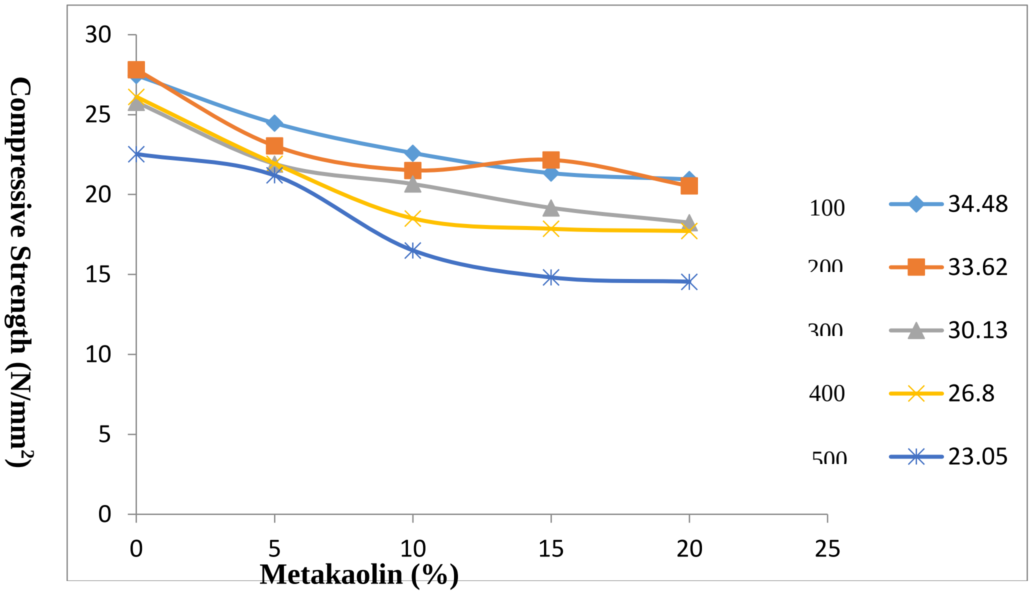

Figure 1. Plot of Compressive Strength versus Metakaolin

Figure 2 shows the plot of compressive strength of OPC-DBP-MTK concrete versus its temperatures. It is observed that the compressive strength of the concrete containing DBP/MTK decreased as the temperature increased irrespective of the replacement levels. For instance, the compressive strength for the control specimen at ambient temperature is 35.75N/mm^2. This value decreased by 3.55%, 5.96%, 15.72%, 25.03% and 32.52% at temperatures of 100^0C, 200^0C,

300^0C, 400^0C and 500^0C respectively. Similarly, (MTK0% DBP 10%) at ambient temperature exhibited a decrease of 4.88%, 3.67%, 10.70%, 9.53% and 21.93% at temperatures of 100^0C,

200^0C, 300^0C, 400^0C and 500^0C respectively. (MTK5% DBP 10%) at ambient temperature decreased by 0.69%, 6.45%, 11.04%, 10.96% and 13.96% at temperature of 100^0C, 200^0C, 300^0C, 400^0C and 500^0C respectively. The compressive strength (MTK 10% DBP10%) at 25^0C decreased by 3.17%, 7.80%, 11.48%, 20.69% and 29.26% at temperatures of 100^0C, 200^0C, 300^0C, 400^0C and 500^0C respectively. Finally, the compressive strength (MTK 20% DBP10%) at 25^0C decreased by 7.59%, 9.40%, 19.40%, 21.84% and 36.26% at temperatures of 100^0C, 200^0C, 300^0C, 400^0C and 500^0C respectively. From these values, the percentage decrease in strength for all percentages of MTK used is minimal from 25^0C to 400^0C, which is one of the properties of a pozzolana that reduces the loss in strength with temperature (Sule, *etal*., 2014). The increase in compressive strength of the blended concrete recorded at 400^0C and above could be attributed to the pozzolanic reaction which led to the formation of additional amount of hydration product (Morsy, *et al.*, 2009). However, the percentage decrease in strength was more significant after 400^0C.

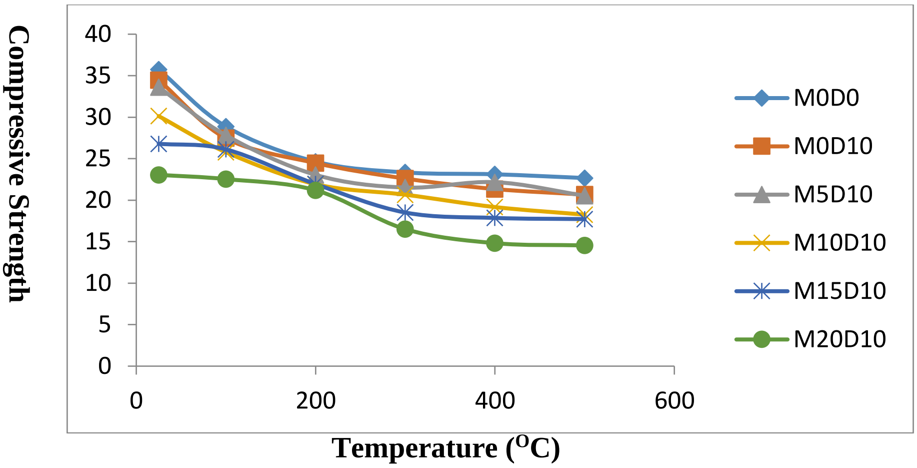

Figure 2. Plot of Compressive Strength versus Temperature

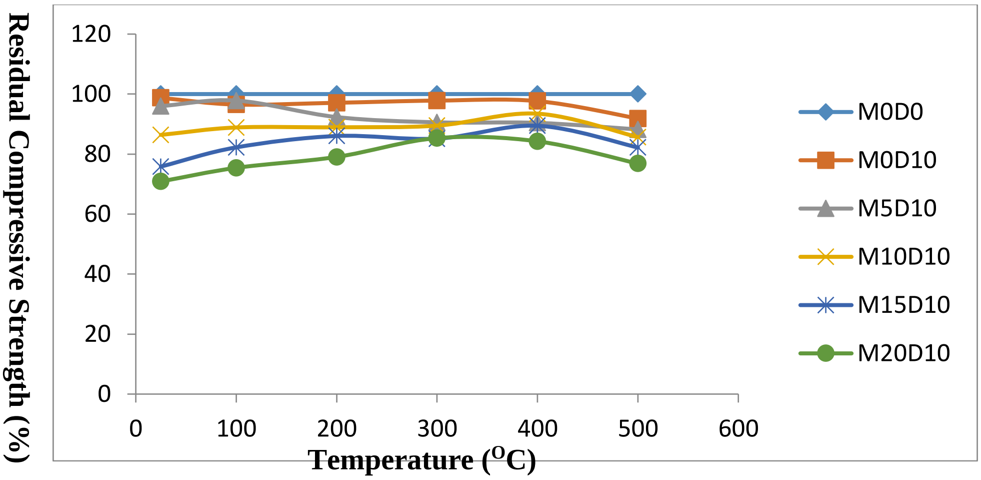

Figure 3. Plot of Residual Compressive versus Temperature

Figure 3 shows the residual compressive strength of each specimen at different temperature. It shows a relative decrease in compressive strength of each specimen thermally treated as compared to its original compressive strength before heating. A distinct pattern of strength loss was observed. Constant compressive strength, slow and steady strength losses were also observed. The constant nature of strength observed may be attributed to low thermal conductivity and very high specific heat capacity of concrete (Sule, et al., 2014). The gradual reduction in strength may be attributed to the pozzolanic reaction of DBP/MTK with cement in concrete and hydrothermal interaction between the cement particles and the embedded pozzolanic materials (Ming-Zhi, et al., 2015). The decrease in compressive strength was observed which may be attributed to the decomposition of Calcium Silicate Hydrate (C-S-H) gel.

3.5 Flexural Strength Test Results

Table 8. Flexural Strength Test Results for the Blended concrete at Different Temperatures.

%Mix	25⁰C	100⁰C	200⁰C	300⁰C	400⁰C	500⁰C
MTK0 DBP 0	6.25	6.17	6.00	5.40	4.74	4.43
MTK0 DBP 10	6.02	5.81	5.89	5.35	4.95	4.5
MTK5 DBP 10	5.87	5.70	5.42	5.22	5.05	4.64
MTK10 DBP 10	5.52	5.40	5.00	4.94	4.70	4.71
MTK15 DBP 10	5.21	5.00	4.71	4.87	4.66	4.39
MTK20 DBP 10	4.66	4.28	4.11	4.00	3.83	3.58

Figure 4 shows the plot of flexural strength of OPC-DBP and MTK concrete versus percentages of MTK used to replace cement. It can be seen from the Figure that generally the flexural strength decreased as the percentages of MTK increased. At temperatures of 25^0C to 500^0C, the flexural strength decreased directly with increase in MTK content. This could be attributed to the replacement of cement with MTK in concrete which results in the reduction of tri-calcium silicates (C_3S), the main strength controlling compound, the loss of moisture which prevent long term hydration and the destruction of an active strength generating ingredient like cement and aggregate due to continuous rise in temperature (Chandam, et al., 2013).

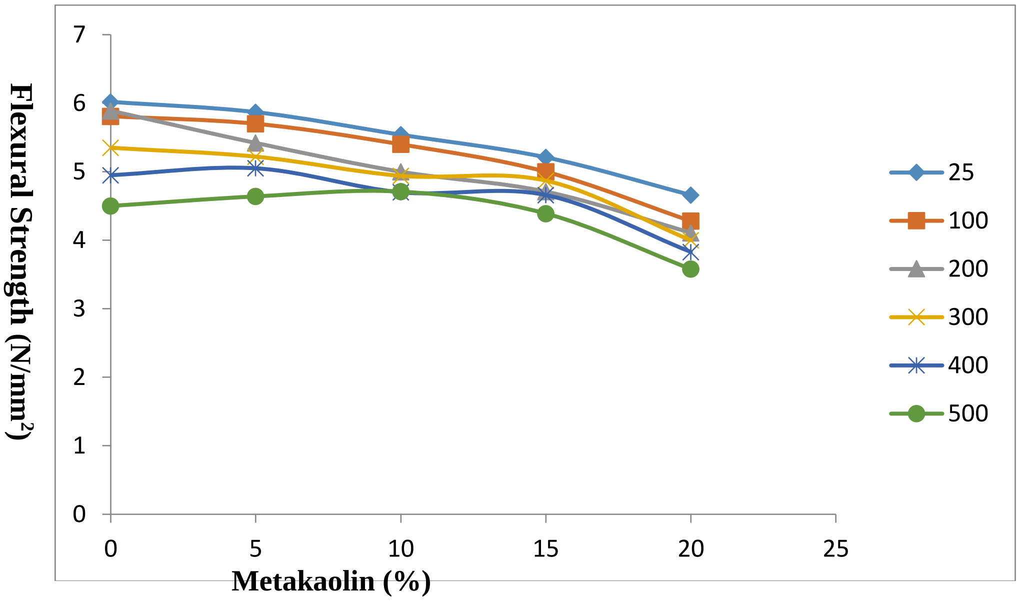

Figure 4. Plot of Flexural Strength versus Metakaolin

Fig 5 shows the plot of flexural strength of OPC-DBP-MTK concrete versus its temperatures. It can be observed that the flexural strength of the concrete containing DBP/MTK decreased as the temperature increased irrespective of the replacement levels. For instance, the flexural strength for the control specimen at ambient temperature is 6.25N/mm^2. This value decreased by 1.28%, 4.0%, 13.6%, 24% and 29.12% at temperatures of 100^0C, 200^0C, 300^0C, 400^0C and 500^0C respectively. Similarly, (MTK 0% DBP 10%) at ambient temperature showed a decrease of 3.49%, 2.16%, 11.13%, 17.78% and 24.58% at temperatures of 100^0C, 200^0C, 300^0C, 400^0C and 500^0C respectively. (MTK 5% DBP10% at ambient temperature decreased by 2.90%, 7.7%, 11.07%, 13.46% and 20.95% at temperatures of 100^0C, 200^0C, 300^0C, 400^0C and 500^0C respectively. The flexural strength (MTK 10% DBP10%) at 25^0C decreased by 2.17%, 9.45%, 10.51%, 14.86% and

14.67% at temperature of 100^{0}C, 200^{0}C, 300^{0}C, 400^{0}C and 500^{0}C respectively. Finally, the flexural strength (MTK 20% DBP10%) at 25^{0}C decreased by 8.37%, 11.80%, 14.16%, 17.81% and 23.18% at temperature of 100^{0}C, 200^{0}C, 300^{0}C, 400^{0}C and 500^{0}C respectively. From the values presented, the percentage decrease in strength for all percentages of MTK used is minimal from 25^{0}C to 400^{0}C, The increase in flexural strength of the blended concrete recorded at 400^{0}C.

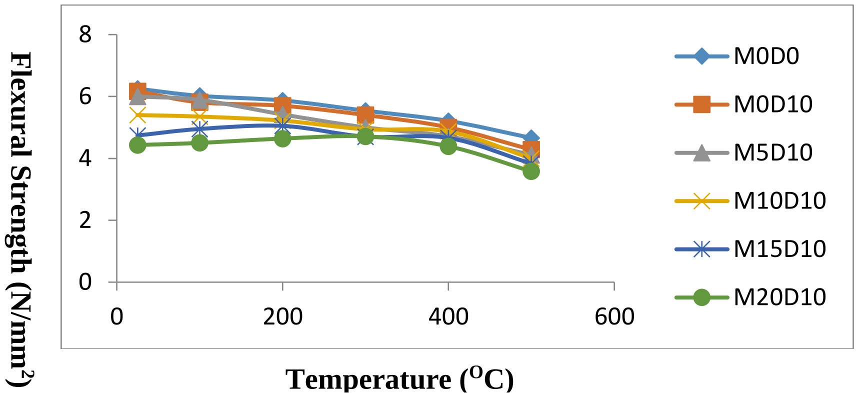

Figure 5. Plot of Flexural Strength versus temperature

Figure 6 shows the residual flexural strength of each specimen at different temperatures. It shows a relative decrease in flexural strength of each specimen thermally treated as compared to its original flexural strength before heating. A distinct pattern of strength loss was observed. Constant flexural strength, slow and steady strength observed may be attributed to low thermal conductivity and very high specific heat capacity of concrete (Sule, et al., 2014). The gradual reduction in strength may be attributed to the pozzolanic reaction of DBP/MTK with cement in concrete and hydro-thermal interaction between the cement particles and embedded pozzolanic materials (Ming-Zhi, et al., 2015). The decrease in flexural strength was observed which may be attributable to the decomposition of calcium silicate Hydrate (C-S-H) gel.

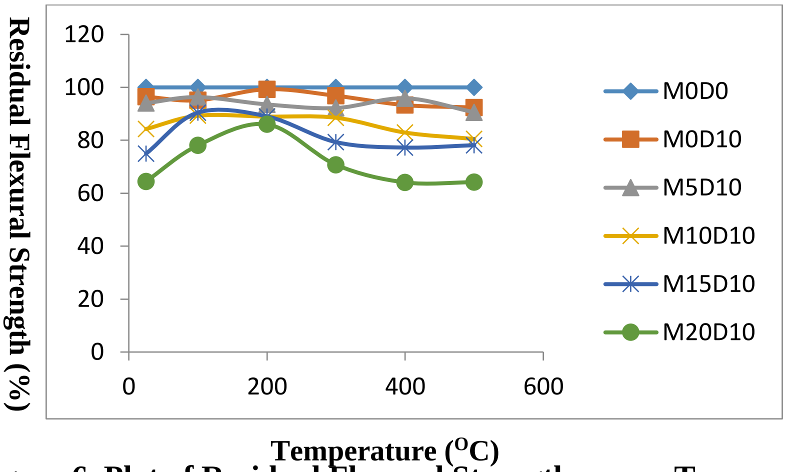

Figure 6. Plot of Residual Flexural Strength versus Temperature

Table 9. Residual Flexural Strength Test Results obtained for OPC-MTKC-DBPC

Temp (^{O}C) / Replacement. level (%)	Ambient Temp 25^{0}C		100^{0}C		200^{0}C		300^{0}C		400^{0}C		500^{0}C	
	F.S	**R.F.S**	**F.S**	**R.F.S**	**F.S**	**R.F.S**	**F.S**	**R.F.S**	**F.S**	**R.F.S**	**F.S**	**R.F.S**
MTK0 DBP0	6.25	100	6.17	98.72	6.00	96.00	5.40	86.40	4.74	75.84	4.43	70.88
MTK0 DBP10	6.02	100	5.18	96.51	5.89	97.84	5.35	88.87	4.95	82.23	5.54	75.42
MTK 5 DBP10	5.87	100	5.70	97.10	5.42	92.33	5.22	88.93	5.05	86.03	4.64	79.05
MTK 10 DBP10	5.22	100	5.40	97.83	5.00	90.58	4.94	89.49	4.70	85.14	4.71	85.33
MTK 15 DBP10	5.21	100	5.00	97.66	4.71	90.40	4.87	93.47	4.66	89.44	4.39	84.26
MTK 20 DBP10	4.66	100	4.28	91.85	4.11	88.20	4.00	85.64	3.83	82.19	3.58	76.82

Table 10. Residual Flexural Strength Test Results obtained for OPC-MTKC-DBPC

Temp (^{O}C) / Replacement level (%)	Ambient Temp. 25^{0}C		100^{0}C		200^{0}C		300^{0}C		400^{0}C		500^{0}C	
	C.S	**R.C.S**	**C.S**	**R.C.S**	**C.S**	**R.C.S**	**C.S**	**R.C.S**	**C.S**	**R.C.S**	**C.S**	**R.C.S**
MTK0 DBP0	35.75	100	34.48	96.45	33.62	94.04	30.13	84.28	26.80	74.97	23.05	64.48
MTK0 DBP10	28.87	100	27.46	95.12	27.81	96.33	25.78	89.30	26.12	90.47	23.54	78.07
MTK 5 DBP10	24.64	100	24.47	99.31	23.05	93.55	21.92	88.96	21.94	89.04	21.20	86.04
MTK 10 DBP10	23.34	100	22.60	96.83	21.52	92.20	20.66	88.52	18.51	79.31	16.51	70.74
MTK 15 DBP10	23.12	100	21.34	93.30	22.17	95.89	19.17	82.92	17.86	77.25	14.82	64.10
MTK 20 DBP10	22.67	100	20.95	92.41	20.54	90.60	18.25	80.50	12.72	78.16	14.55	64.18

3.6. Compressive Strength Statistical Analysis

The analysis was carried out using response surface regression i.e. compressive strength versus percentage (%) mix, temperature. The regression equation as given by Minitab Software is

$$\mathbf{Cs = 32.4205 - 1.30656A - 0.00671399B + 0.043510A^2 - 1.80776E - 05B^2 + 0.000101991A} \quad (3)$$

Where A and B (independent predictor variables) are percentage (%) mix and temperature respectively, while Cs is the compressive strength (response variable) in the model equation. If all the terms are set to zero, the compressive strength, Cs value will be 32.4205N/mm^2.

Table 11. Estimated Regression Coefficients for Compressive Strength

Predictor	Coefficient	SE coefficient	T	P
Constant	20.6497	0.5762	5.835	0.000
Percentage (%) Mix (A)	-4.7276	0.3813	-12.399	0.000
Temperature (B)	-3.6064	0.4040	-8.926	0.000
% mix x %mix (A^2)	4.0351	0.6944	5.811	0.000

Temperature x temperature (B^2)	-0.0197	0.6947	-1.468	0.153
% Mix x temperature (AB)	0.2422	0.5438	0.445	0.659

LEGEND: SE =Standard error; T = t-test; P = Probability

Since the quadratic terms i.e. the highest power of the factor to factor relationship, R^2 produced the lowest predicted residual error sum of square (PRESS) value of 119.530, highest number of terms that are statistically significant i.e. ($\rho\ value < 0.05$), highest amount of variation in the data explained by the model, R^2 (adjusted) = 92.33%, highest number of predictions explained by the model R^2 (predictor) = 96.57% are within 20% of the adjusted R^2 which shows that the

model is not over fit neither too complex i.e. R^2 (adjusted) - R^2 (Predicted) < 20%. Based on this, quadratic model, R^2 was recommended by Minitab Software for performing estimated coefficient and predictions for compressive strength. The statistical significance for the compressive strength was checked using the analysis of variance (ANOVA). Table 12 shows the analysis of variance of compressive strength.

Table 12. Analysis of Variance of Compressive Strength

Source	DF	Seq. SS	Adj. SS	Adj. MS	F	P
Regression	5	808.692	808.692	161.738	59.60	0.000
Linear	2	710.666	627.358	313.679	115.60	0.000
%Mix	1	482.294	417.142	417.142	153.72	0.000
Temp	1	228.372	216.220	216.220	79.68	0.000
Square	2	97.488	97.488	48.744	17.96	0.005
%Mix %mix	1	91.641	91.641	91.641	33.77	0.000
Temp x Temp	1	5.846	5.846	5.846	2.15	0.153
Interaction	1	0.538	0.538	0.538	0.20	0.659
%mix x Temp	1	0.538	0.538	0.538	0.20	0.659
Residua/Error	30	81.408	81.408	2.714	-	-
Total	35	890.100	-	-	-	-

LEGEND: DF = Degree of freedom; Seq. SS = Sequential sums of squares; Adj. SS = Adjusted sums of squares; Adj. MS = Adjusted mean squares; F = F-test or distribution; P = Probability.

The P-value is a number describing how likely it is that data generated have occurred under null hypothesis of the statistical test. If p-value is less than 0.05, the data are said to be statistically significant. Thus, from the ANOVA table, the overall regression model p-value is less than the level of significance (0.05). Therefore the model hypothesis is statistically significant. The p-value for the linear term for both factors (percentage mix and temperature) are also lower than the level of significant. Therefore, the linear terms significantly affect compressive strength of the material. Also, the interaction between the %mix and temperature was observed not to be statistically significant with respect to the compressive strength since the p-value is 0.659.
Consequently, the p-value for the square terms (quadratic) is also observed to be lower than the level of significance, thus they possess a significant effect on the compressive strength of the material.

3.7 Flexural Strength Statistical Analysis

The analysis was also carried out using response surface regression i.e. flexural strength versus % mix and temperature. The regression equation as given by Minitab Software is

$$\mathbf{Fs=6.26896-0.030043A-0.00283589B-0.00310623A^2-1.03427E-06B^2+0.0001483AB} \quad (4)$$

Where A and B (independent predictor variables are percentage (%) mix and temperature respectively, while Fs is the flexural strength (response variable) in the model equation. If all the terms are set to zero, the flexural strength, Fs value will be 6.26896N/mm^2.

Table 13. Estimated Regression Coefficients for Flexural Strength.

Predictor	**Coefficient**	**SE coefficient**	**T**	**P**
Constant	5.10860	0.05843	87.434	0.000
%mix (A)	-0.65529	0.03866	-16.949	0.000
Temp (B)	-0.56146	0.04096	-13.706	0.000
%mix x %mix(A^2)	-0.31062	0.07040	-4.412	0.000
Temp x temp (B^2)	-0.05834	0.07044	-0.828	0.414
% mix x temp (AB)	0.24102	0.05514	4.371	0.000

LEGEND: SE =Standard error; T = t-test; P = Probability

Since the quadratic model produced the lowest predicted residual error sum of square (PRESS) value of 1.29177, highest number of terms that are statistically significant (i.e. terms with p-value < 0.05), highest amount of variance in the data explained by the model (R^2(adj) = 93.87%, highest number of predictions explained by the model (R^2(pred) = 91.89% are within 20% of the adjusted R^2 to show that the model is not over fit neither too complex i.e. R^2 (adj) – R^2 (predicted) < 20%. Therefore, the quadratic model was recommended by Minitab software for performing estimated coefficient and predictions for flexural strength of materials. The statistical significance for the flexural strength was also checked using the analysis of variance (ANOVA) table probability value, p-value. Table 4.30 shows the analysis of variance of flexural strength.

Table 14. Analysis of Variance of Flexural Strength

Source	**DF**	**Seq SS**	**Adj SS**	**Adj MS**	**F**	**P**
Regression	5	15.0994	15.0994	3.01987	108.25	0.000
Linear	2	14.0041	13.1253	6.56263	235.23	0.000
% mix	1	7.8272	8.0141	8.01415	287.26	0.000
Temperature	1	6.1769	5.2407	5.24073	187.85	0.000
Square	2	0.5622	0.5622	0.28110	10.08	0.000
%mix x mix	1	0.5431	0.5431	0.54306	19.47	0.000
Temp x temp	1	0.0191	0.0191	0.01914	0.69	0.414
Interaction	1	0.5331	0.5331	0.53306	19.11	0.004
%mix x temp	1	0.5331	0.5331	0.53306	19.11	0.000
Residue /Error	30	0.8369	0.8369	0.02790	-	-
Total	35	15.9363	-	-	-	-

LEGEND: DF = Degree of freedom; Seq. SS = Sequential sums of squares; Adj. SS = Adjusted sums of squares; Adj. MS = Adjusted mean squares; F = F-test or distribution; P = Probability.

The p-value is a number describing how it is that data generated would have occurred under null hypothesis of the statistical test.If p-value is less than 0.05, the data are said to be statistically significant. Thus, from the ANOVA table, the overall regression model p-value is less than the level of significant (0.05). Therefore, the model hypothesis is statistically significant. Thus, the p-value for the linear terms for both factors (% mix and temp.) are also lower than the level of significant. Also, the interaction between the % mix and temperature was observed to be statistically significant with respect to the flexural strength since the p-value is 0.004. Consequently, the p-value for the square terms (quadratic) is also observed to be lower than the level of significance, thus, they statistically affect the flexural strength of the materials.

3.8 Validation of the Developed Model Equation for Compressive Strength

It is necessary to check the validity of the model equation developed before drawing conclusions. There are two basic assumptions in ANOVA analysis: Normality and constant variance. The normality assumption is satisfied if the distribution of the residuals aligns closely to the straight line drawn in the plot. It can be seen that the residuals (dots) in Fig 4.9 align themselves very closely and tend to resemble a strength line in the probability plot (normal probability plot) indicating that there is a normal distribution for the residuals. Hence, the normality assumption is satisfied (John, 2013).

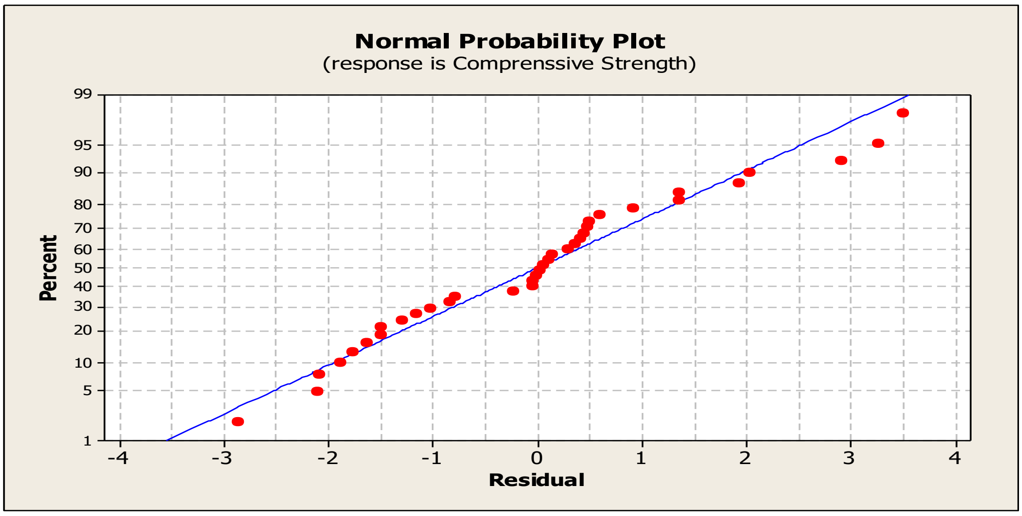

Figure 7. Normal Probability plot for Compressive strength

Furthermore, contour (heat map) is used to explain relationship between the response (compressive strength) and the variables (% mix and temp.). From Fig 4.10, the minimum compressive strength was obtained between 15% - 20% mixture and within 400^{0}C where as the maximum compressive strength was observed at 0%-3% mixture and at 0^{0}C - 300^{0}C

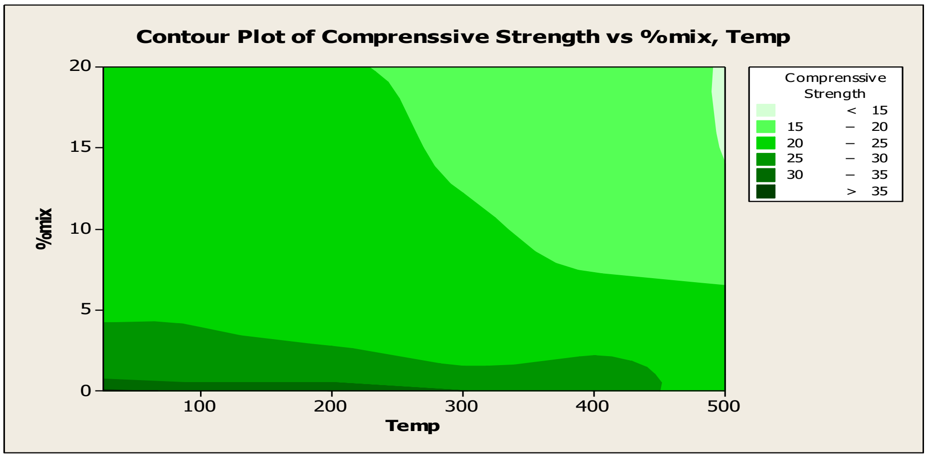

Figure 8. Contour plot of Compressive strength Vs % Mix and Temperature

3.9 Relationship between Actual versus Predicted Value for Compressive Strength.

From Figure 9, there is a perfect correlation between the actual values and the predicted values, which signifies that there is a very good agreement between the model predictions and experimental results.

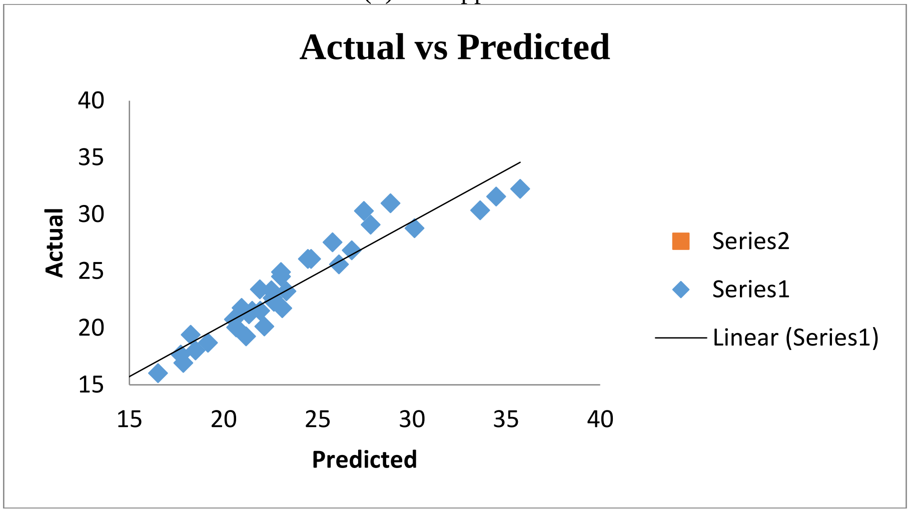

Figure 9. Relationship between Actual versus Predicted Value for Compressive Strength

3.10 Validation of the Developed Model Equation for Flexural Strength.

The normality assumption is also satisfied if the distributions of the residuals align closely to the straight line drawn in the plot. It can be seen that the residual clots in Fig. 4.12 aligns themselves very closely and tend to resemble a strength line in the probability plot (normal probability plot) indicating that there is a normal distribution for the residuals. Hence, the normality assumption is satisfied (John, 2013).

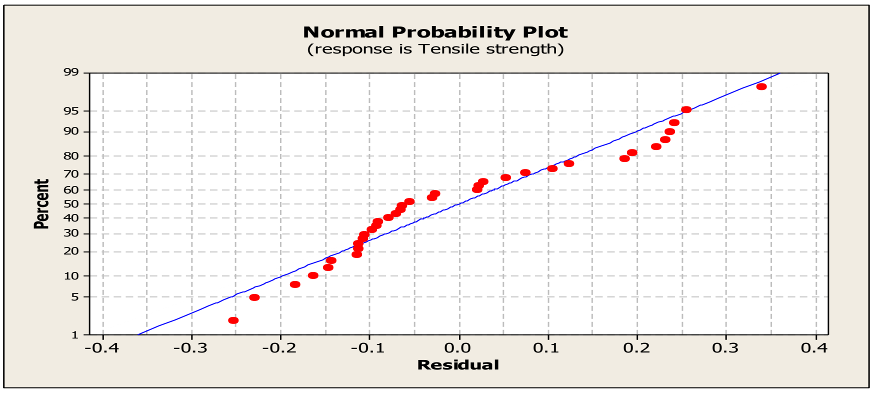

Figure 10. Normal Probability plot for Tensile Strength

Furthermore, contour (heat map) is used to explain the relationships between the response (flexural strength) and the variables (% mix and temp). From Fig.4.13, the minimum flexural strength was obtained between 18%-20% mixture and with 350^{0}C whereas the maximum flexural strength was observed at 0% -3% mixtures and at 0^{0}C.

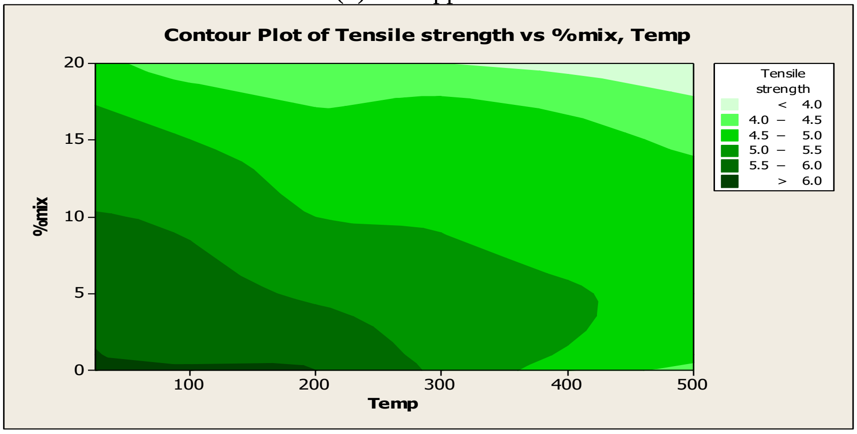

Figure 11. Contour plot of Tensile Strength Vs %Mix and Temp

3.11 Relationship between Actual versus Predicted Values for Flexural Strength

From Figure 12, there is a perfect correlation between the actual values and the predicted values, which signifies that there is a very good agreement with the model predictions and experimental results for the flexural strength.

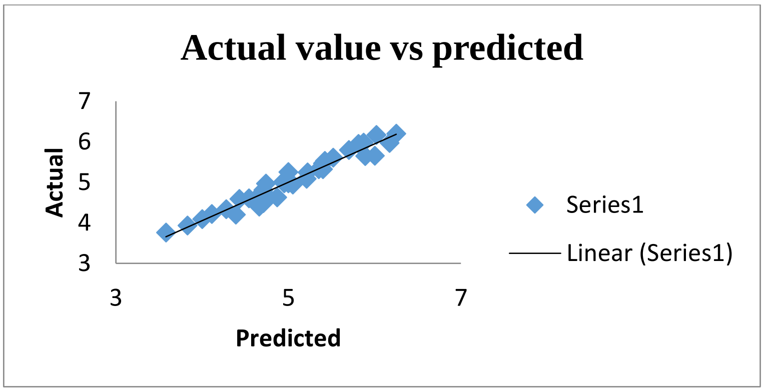

Figure 12. Relationship between Actual versus Predicted Value for Tensile Strength

4. Conclusion

From the results obtained, the following conclusions were made:

i. The discarded bottle powder (DBP) and metakaolin (MTK) are classified as class N pozzolana according to ASTMC 618 (2012) specification.
ii. The workability of the concrete is strongly affected when DBP and MTK are introduced which necessitates the increase in the quantity of water used to maintain uniform mix.
iii. Compressive and tensile strengths generally decreased with high temperatures.
iv. The statistical models developed using response surface regression and analyses of variance (ANOVA) indicate that there was no statistical significant difference between the experimental and predicted strength value at 0.05 level of significant.
v. Percentage mixes and temperatures were useful independent predictors of the regression model for the response variable (compressive strength).
vi. The regression model equation predicted from independent variables (A = % mix, B = Temperature) is: compressive strength,

$$\mathbf{Cs = 32.4205 - 1.30656A - 0.00671399B + 0.0403510A^2 - 1.80776E\ {-05}B^2\ 0.00010991AB}$$

where the coefficient of determination, $R^2 = 96.57\%$, A and B (independent predictor variables) are percentage mixes (%) and temperatures respectively.

vii. The regression model equation predicted from the independent variables (A = % mix and B = temperature) is: flexural strength,

$$\mathbf{Fs= 6.26896-0.0300434A - 0.00283589B - 0.0031623A^2 -1.03427E - 06B^2 + 0.0001483AB}$$

and the coefficient of determination, $R^2 = 91.89\%$, where A and B (independent predictor variables) are percentage (%) mixes and temperatures respectively.

Abbreviation and Symbols

^{0}C	Degree Celsius
a_1, $a_{2\cdots f1f2}$	Concrete cube and beams identification number
A	Metakaolin
Adj SS	Adjusted sums of squares
ANOVA	Analysis of variance
Al_2O_3	Aluminum oxide
ASR	Alkali Silica Reaction
ASTM	American Society of Testing Materials
B	Temperature
BS	British Standard
CaO	Calcium oxide
C_2S	Dicalcuim Silicate
C_3A	Tricalcium Aluminate
C_3S	Tricalcium Silicate
C_3AF	Tricalcium Alumino Ferrite
C-A-H	Tricalcium Aluminum Hydrogen
CO_2	Carbon Dioxide
Coeff.	Coefficient
C_S	Compressive strength
CSF	Condensed Silicate Hydrogen
DF	Degree of Freedom for Regression
DBP	Discarded Bottle Powder
F	Fisher F-Test Calculated from Regression
Fe_2O_3	Iron oxide
FA	Fly Ash
F_S	Flexural strength
KN/mm^2	Kilo Newton per Millimeter square
Kg/m^3	Kilogram per cubic meter
LOI	Loss on Ignition
MgO	Magnesium oxide
MTK	Metakaolin
mm	Millimeter
MOR	Modulus of Rupture
Mpa	Mega Pascal
MS	Mean of squares for Regression
OPC	Ordinary Porttand cement
P	Probability of test statistics
POFA	Palm Oil Fuel Ash
PVC	Poly Vinyl Chloride
R.F.S.	Residual Flexural strength
R^2	Coefficient of Determination
R.C.S	Residual Compressive Strength
RHA	Rice Husk Ask
SCMs	Supplementary Cementituous Materials
SiO_2	Silicon oxide
SECoeff	Standard Error Coefficient
SE	Standard Error
SF	Silica Flour
SeqSS	Sequential Sums of Squares
T	Student t-Test from regression

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