

Mixture Experiment Design for the Workability of High Performance Recycled Coarse Aggregate Concrete

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ABSTRACT:

This study is on mixture experiment design of the workability of high performance recycled coarse aggregate concrete, (i.e. high performance concrete produced with recycled coarse aggregate). slump test, which is a widely used test for determining workability of concrete, was the response property used to study the workability of the high performance concrete (HPC) produced in this work. The following components constituted the HPC mixture: water, cement, silica fume, high range water reducing admixture, natural coarse aggregate, recycled coarse aggregate, and fine aggregate. The optimization procedure followed the creation of a model for the slump of the high performance concrete. The model was based on the response surface methodology, using Scheffe's quadratic polynomial for mixture experiments. The mixture experiment design and analysis were performed using MINITAB 17 statistical software. 46 different mixture experiments were performed. The results of the mixture experiments were used to develop a regression model for slump using analysis of variance (ANOVA) and least squares principles. The developed model was applied to carry out a numerical optimization of the HPC using the desirability function procedure. The range of predictable values for slump obtained from experiments in this work varied from 80 mm to 170 mm, and the model could predict and optimize the response property within this range.

KEY WORDS: Model; High performance concrete; recycled coarse aggregate; High range water reducing admixture (Superplasticizer), Extreme vertices design, Mixture Experiment design.

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I. INTRODUCTION

One of the properties of HPC required to meet some "special combinations of performance" is usually its high workability. According to [1], workability of concrete is defined in ASTM C-125 as the property determining the effort required to manipulate a freshly mixed quantity of concrete with minimum loss of homogeneity. [1] also stated that consistency is used as the simple index for mobility or flowability of fresh concrete, and that the slump test is the most universally used test to measure the consistency of concrete. [2] observed that slump within the range of 150 – 200 mm could be achieved for a HPC mix. [3 & 4] concluded that when recycled aggregate is used in the saturated surface dry state to produce new concrete, the consistency of the recycled aggregate concrete does not differ considerably from the comparable conventional concrete, but when dry recycled aggregate is used, an extra amount of water would be needed to achieve the same consistency as the conventional concrete. [5] discovered that when recycled concrete aggregate was coated with a dispersant and used in the production of concrete,

the slump loss with time reduced as the percentage of the coated recycled concrete aggregate (CRCA) in the concrete mix increased, and that the highest slump loss with time was recorded with a 100% recycled concrete aggregate (RCA,) i. e., when no percentage of recycled concrete aggregate in the concrete mixture was coated. The conclusion was that it was due to the high absorption of the RCA.

Since concrete is a mixture of different components, in this work, it was necessary to find a method which will not only be precise in measuring the workability of HPC, but can also produce optimum combination of the mixture component to obtain a desired workability. The mixture experiment approach presented in details by [11], found useful application in this. [12] observed that it is possible to use the extreme vertices design of mixture experiment to optimize the compressive strength of high performance recycled coarse aggregate concrete, using a statistical software (MINITAB 17) [13]. MINITAB 17 provided an easy way of performing complex statistical analysis including ANOVA and the least squares method,

associated with the design and analysis of mixture experiments, as presented in [12].

II. MATERIALS AND METHODS

The materials used in this work include Water, cement, silica fume (micro silica), high range water reducing admixture (HRWRA or superplasticizer), natural coarse aggregate, recycled coarse aggregate, and fine aggregate. The water used was tap water obtained from the strength of materials laboratory of the Cross River State University of Technology, Calabar, for both concrete casting and curing. Ordinary Portland cement was obtained from a local cement supplier and used for all concrete works. Micro Silica (Silica fume) of specific gravity of 2.2 in powdered form; was used. The superplasticizer used for this work is Conplast SP430. Conplast SP430 is a chloride free superplasticising admixture based on selected sulphonated naphthalene polymers. It is supplied as a brown solution which instantly disperses in water [14]. The natural aggregate used is granite of intrusive igneous rock origin, with a maximum particle size of 20mm. The recycled coarse aggregate used was free from impurities and was obtained from a demolished concrete structure site in the Calabar metropolis. It was manually crushed to approximate maximum particle sizes in the range of 20mm – 25mm. The fine aggregate used is river sand and was obtained from the Cross River, through local suppliers. The particle size distribution for the aggregates used in this work was conducted in accordance with [15], Table 2 is a summary of other physical properties of aggregates used in this work, they were conducted in accordance with relevant standards [16& 17].

Extreme vertices design of the mixture experiment approach was used to produce the design points on which experiments were conducted. The workability property obtained from the experiments was modeled on the Scheffe's second degree polynomial for a seven component mixture experiment. [18] proposed the use of $\{q, m\}$ symmetric canonical polynomial models obtained by reparameterization of standard polynomials of degree m , for q components by using equation 2. Since for a mixture experiment, the components are combined in proportions and not amounts, the components proportions must always sum to 1, equation 2 [11]. For a mixture of degree (7, 2), the Scheffe's second degree polynomial model will contain 28 constant terms and the model equation is given by equation (5).

For a standard polynomial;

$$y = \beta_0 + \beta_1x_1 + \beta_2x_2 + \dots + \beta_qx_q + e \quad (1)$$

But for mixture experiment of "q" number of components;

$$x_1 + x_2 + x_3 \dots x_q = 1 \quad (2)$$

Hence, by replacing β_0 with $\beta_0(x_1 + x_2 + \dots + x_q)$, in equation 1, equation 1 can be rewritten to obtain equation (3).

$$y = \beta_1x_1 + \beta_2x_2 + \dots + \beta_qx_q + e \quad (3)$$

Where the term β_i are constants which replaced the sum ($\beta_0 + \beta_i$), x terms are the mixture components proportions and, e is the random error.

Equation 3 is known as Scheffe's linear mixture polynomial or Scheffe's first degree polynomial equation. The Scheffe's quadratic or second degree polynomial equation is written in a similar manner but contains a quadratic part. The quadratic part of the Scheffe's second degree polynomial equation arises as a result of possible interactions between mixture components. Scheffe's second degree polynomial equation is given in equation 4.

$$y = \beta_1x_1 + \beta_2x_2 + \dots + \beta_qx_q + \beta_{12}x_1x_2 + \beta_{13}x_1x_3 + \dots + \beta_{ij}x_ix_j + e \quad (i < j) \quad (4)$$

Now, for a mixture of degree (7, 2), the Scheffe's second degree polynomial equation is as given in equation 5

$$y = \beta_1x_1 + \dots + \beta_7x_7 + \dots + \beta_{12}x_1x_2 + \dots + \beta_{67}x_6x_7 + e \quad (5)$$

x_ix_j are interaction terms, and y is the studied response property.

To develop a model which would eventually be used for optimization, mixture experiment must be conducted, using the seven mixture components earlier listed, on at least 28 different mixes of HPC and the response property of interest (workability) shall be measured. The 28 different mixes correspond to the number of terms (model coefficients) that would be in equation 5. However, to validate the model to be developed, additional mixes of the HPC are required. Hence, in addition to the 28 distinct mixes needed to estimate the twenty eight model coefficients, 10 distinct mixes were added to check the adequacy of the model and 8 mixes from the augmented design were replicated mixes; 5 mixes replicated once to test the statistical significance of the fitted coefficients and 1 other mix replicated once for each week of experiment (three weeks), to check statistical control of the fabrication and measurement process [19], making a total of 46 experimental design points.

In producing a (7, 2) extreme vertices design for the mixture experiment to be conducted, the proportions of the mixture components to be used were determined. The proportions of the mixture components were the expected volume fractions of individual components. A reference mix proportion for the HPC used in the construction of main piers and T-beams of the Confederation Bridge in Canada [1], gave a guide for the selection of possible ranges of volume fractions of the mixture components, these ranges were the upper and lower bounds of volume fractions of the mixture components. The reference mix was a conventional HPC, therefore, the upper and lower bounds were modified to accommodate the recycled coarse aggregate used in this work. The upper and lower bounds of mixture components used in this work are

presented in Table 1. The upper and lower bounds of the natural and recycled coarse aggregates were so selected as to obtain an alternate random distribution of both coarse aggregates in every design point. However, the constraint (0.4 natural coarse aggregate + recycled coarse aggregate 0.44) was imposed on both the natural and recycled coarse aggregates, so as to ensure that the coarse portion of the HPC mixture did not exceed the specify volume fraction for coarse aggregate in any case, as obtained from the reference mix.

These upper and lower bounds of volume fractions of the mixture components were imputed into the “Design mixture experiment” function of MINITAB 17. MINITAB 17 produced a total of 81 candidate design points (in terms of volume fractions) from which a random set of 46 design points were selected. In the 46 selected design points, it was ensured that eight mixes are replicated to check the validity of developed model as earlier described. The volume fractions of the mixture

components were then converted into mass of components for batching, using the specific gravity of individual components. 46 concrete mixes were batched and the slump test which is the most universally used test for measuring the workability of concrete according to [1], was conducted in accordance with [20], under normal condition of temperature.

Table 3 shows the mix proportions in mass of the 46 concrete mixes. The mixture experiment (i.e. the batching and performing of slump test on batched HPC mix) was carried out within a period of three weeks. Each batch of concrete was approximately 0.0032m³ in volume and was mixed manually in a mixing pan. The slump test was conducted with a slump cone of dimensions: Bottom diameter = 200 mm, Top diameter = 100 mm, Height = 300 mm, and an average from two slump tests for each HPC mix was obtained and also presented in Table 3.

Table 1: Upper and Lower bounds of mixture components

Components	ID	Minimum Volume fraction	Maximum Volume fraction
Water	X ₁	0.16	0.185
Cement	X ₂	0.128	0.148
Silica fume	X ₃	0.015	0.029
HRWRA	X ₄	0.0121	0.0401
Natural Coarse aggregate	X ₅	0.060	0.340
Recycled Coarse aggregate	X ₆	0.060	0.340
Fine aggregate	X ₇	0.28	0.3054

The data obtained from the mixture experiments (i.e. the slump test results) were imputed into the “Analyze mixture experiment” function of MINITAB 17. MINITAB 17 analyzed the data and fitted the data to a quadratic model with an initial 28 terms. However, using test of terms significance as obtained from [12], it was observed that not all the 28 model terms in the initial quadratic model were statistically significant to be included in the model, hence, the initial model was reduced to the model given in equation (6). The final selected model was then used to obtain optimum component proportions that would produce

a desired workability. The optimization procedure was performed using the “Optimization” function of MINITAB 17 as also obtained from [12].

III. RESULTS

In the presentation of mixture experiment results, the following variables x₁, x₂, x₃, x₄, x₅, x₆, and x₇ were used to represent the mixture component proportions for Water, Cement, silica fume, HRWRA, natural coarse aggregate, recycled coarse aggregate, and fine aggregate respectively.

Table 2: summary of other physical properties of aggregate

Physical Properties	Aggregate	
	Natural	Recycled
Specific Gravity	2.8	2.49
Aggregate Crushing Value (%)	23.64	28.91
Aggregate Impact Value (%)	20.73	25.17
Moisture Absorption Value (%)	0.0	4.6

Table 3: Batching weights of mixture components and results for average Slump

Std Order	Run Order	Water (kg/m ³)	Cement (kg/m ³)	Silica Fume (kg/m ³)	HRWRA (l/m ³)	Natural Coarse agg. (kg/m ³)	Recycled Coarse agg.(kg/m ³)	Fine agg. (kg/m ³)	Slump(mm)
2	1	151.8	382.6	41.5	11.5	159.4	803.3	728.0	120.0
67	2	151.8	382.6	31.3	11.7	479.8	518.5	740.1	130.0
78	3	152.2	391.1	32.2	11.9	717.9	307.6	729.1	127.5
80	4	152.2	383.8	32.2	11.9	717.9	307.6	735.4	120.0
72	5	152.2	391.1	32.2	11.9	345.9	638.4	729.1	110.0
26	6	151.8	382.6	41.5	11.5	531.4	472.6	728.0	145.0
47	7	154.2	382.6	31.3	11.5	903.4	141.8	734.4	130.0
10	8	156.5	382.6	31.3	11.5	903.4	141.8	728.0	137.5
46	9	154.2	382.6	31.3	11.5	159.4	803.3	734.4	102.5
27	10	152.2	383.8	32.2	11.9	352.5	638.4	729.1	100.0
17	11	151.8	382.6	31.3	13.8	159.4	803.3	734.4	91.4
69	12	152.6	385.0	33.0	12.3	532.5	473.5	730.1	115.5
5	13	151.8	382.6	31.3	11.5	159.4	803.3	740.7	120.0
21	14	151.8	382.6	31.3	16.1	531.4	472.6	728.0	120.0
35	15	151.8	397.2	31.3	11.5	531.4	472.6	728.0	145.0
4	16	156.5	382.6	31.3	11.5	159.4	803.3	728.0	95.0
7	17	151.8	382.6	31.3	11.5	172.4	803.3	728.0	80.0
73	18	154.5	383.8	32.2	11.9	345.9	638.4	729.1	115.0
74	19	152.2	383.8	32.2	11.9	345.9	638.4	735.4	110.0
17	20	151.8	382.6	31.3	13.8	159.4	803.3	734.4	90.5
60	21	153.4	382.6	34.7	13.0	903.4	141.8	728.0	150.0
8	22	151.8	382.6	41.5	11.5	903.4	141.8	728.0	170.0
34	23	151.8	389.9	31.3	11.5	165.9	803.3	728.0	110.0
1	24	151.8	382.6	31.3	11.5	903.4	153.3	728.0	115.0
16	25	151.8	382.6	31.3	11.5	531.4	472.6	740.7	145.0
72	26	152.2	391.1	32.2	11.9	345.9	638.4	729.1	107.5
12	27	151.8	382.6	31.3	16.1	903.4	141.8	728.0	127.5
75	28	152.2	383.8	32.2	14.2	345.9	638.4	729.1	97.5
22	29	151.8	382.6	36.4	11.5	159.4	803.3	734.4	102.5
38	30	151.8	382.6	31.3	13.8	903.4	141.8	734.4	145.0

Std Order	Run Order	Water (kg/m ³)	Cement (kg/m ³)	Silica Fume (kg/m ³)	HRWR A (l/m ³)	Natural Coarse agg. (kg/m ³)	Recycled Coarse agg.(kg/m ³)	Fine agg. (kg/m ³)	Slump(mm)
11	31	151.8	382.6	31.3	11.5	903.4	141.8	740.7	152.5
69	32	152.6	385.0	33.0	12.3	532.5	473.5	730.1	115.5
73	33	154.5	383.8	32.2	11.9	345.9	638.4	729.1	125.0
8	34	151.8	382.6	41.5	11.5	903.4	141.8	728.0	160.0
79	35	154.5	383.8	32.2	11.9	717.9	307.6	729.1	140.0
35	36	151.8	397.2	31.3	11.5	531.4	472.6	728.0	125.0

3	37	151.8	397.2	31.3	11.5	159.4	803.3	728.0	117.5
50	38	156.5	382.6	31.3	11.5	531.4	472.6	728.0	117.5
70	39	152.2	383.8	32.2	11.9	717.9	313.4	729.1	110.0
69	40	152.6	385.0	33.0	12.3	532.5	473.5	730.1	100.0
69	41	152.6	385.0	33.0	12.3	532.5	473.5	730.1	100.0
9	42	151.8	397.2	31.3	11.5	903.4	141.8	728.0	145.0
51	43	153.4	382.6	34.7	13.0	159.4	803.3	728.0	95.0
24	44	152.2	383.8	32.2	14.2	717.9	307.6	729.1	120.0
77	45	152.2	383.8	37.3	11.9	717.9	307.6	729.1	95.0
33	46	151.8	389.9	31.3	11.5	903.4	147.6	728.0	125.0
Ref 1	0%	153	416	34	11.4	1030	0	737	185
Ref 2	100%	153	416	34	11.4	0	1030	737	185

Table 4: Final regression analysis for Slump

Estimated Regression Coefficients for SLUMP(mm) (component proportions)						
Term	Coefficients	SE Coef	T	P	VIF	
X ₁	-1966225	437870	*	*	4395750206	
X ₂	-2132746	516084	*	*	3921021580	
X ₃	7087461	1006876	*	*	224660793	
X ₄	246436	78908	*	*	920179	
X ₅	248676	78833	*	*	281304401	
X ₆	248799	78831	*	*	276347345	
X ₇	446914	85165	*	*	507403907	
X ₁ * X ₂	17330515	4023008	4.31	0	6165279798	
X ₂ * X ₃	-25587799	5163330	-4.96	0	98070535	
X ₃ * X ₇	-12692418	4037156	-3.14	0.003	285043839	
X ₄ * X ₅	20164	8656	2.33	0.026	571	
S = 7.12695		PRESS = 3315.33				
R-Sq = 90.46%		R-Sq(pred) = 81.69%		R-Sq(adj) = 87.66%		
Analysis of Variance for SLUMP(mm) (component proportions)						
Source	DF	Seq SS	Adj SS	Adj MS	F	P
Regression	10	16379.4	16379.4	1637.94	32.25	0
Linear	6	12689.6	6325.8	1054.31	20.76	0
Quadratic	4	3689.8	3689.8	922.45	18.16	0
X ₁ * X ₂	1	61.3	942.6	942.6	18.56	0
X ₂ * X ₃	1	2854.3	1247.4	1247.42	24.56	0
X ₃ * X ₇	1	498.6	502	502.05	9.88	0.003
X ₄ * X ₅	1	275.6	275.6	275.62	5.43	0.026
Residual Error	34	1727	1727	50.79		
Lack-of-Fit	26	1183.2	1183.2	45.51	0.67	0.792
Pure Error	8	543.8	543.8	67.97		
Total	44	18106.4				

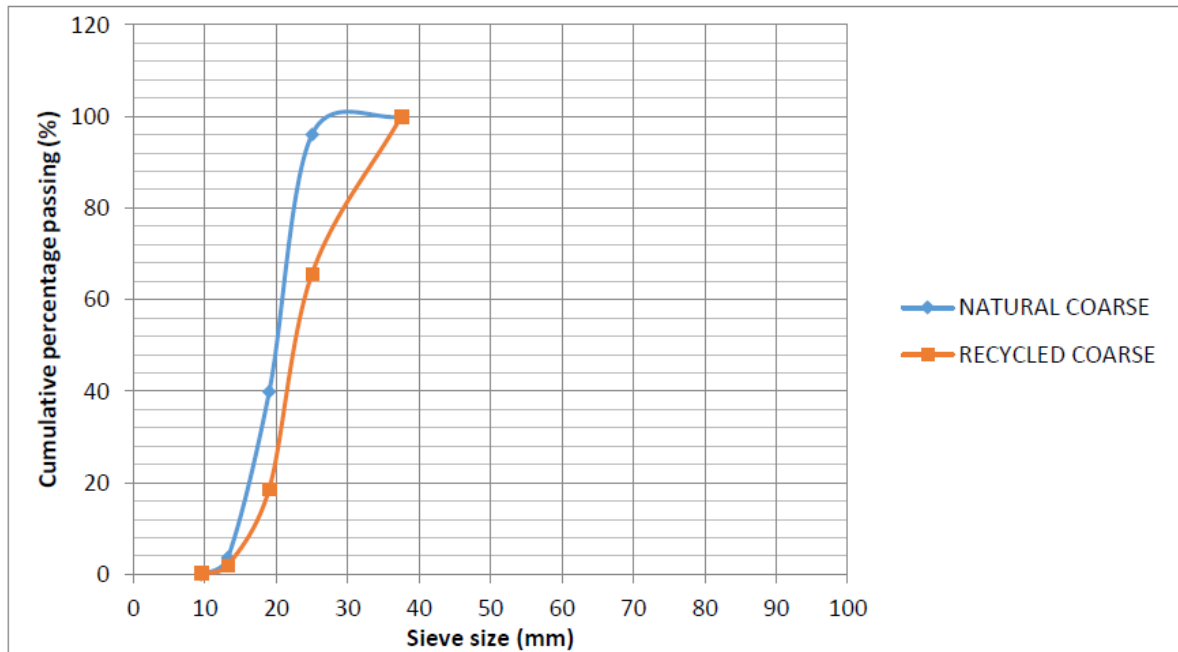


Figure 1: Gradation curves of natural and recycled coarse aggregates

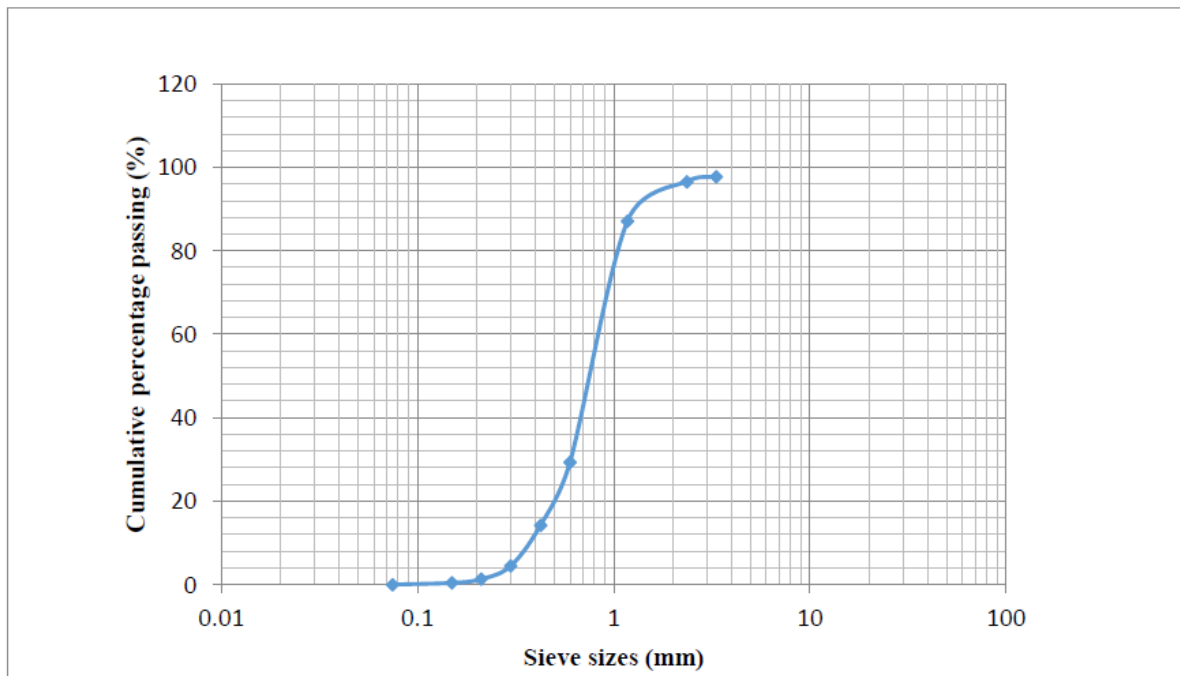


Figure 2: Gradation curve of fine aggregate

IV. DISCUSSION

The results in Table 2 show that the aggregate crushing values and aggregate impact values of both natural and recycled coarse aggregates meet the minimum specification of [21], which is that both aggregate crushing value and aggregate impact value of coarse aggregates for structural concrete should not be greater than 30%. For the moisture absorption of the two coarse

aggregates in Table 2, the recycled coarse aggregate with a moisture absorption value of 4.6% indicates that the recycled coarse aggregate is likely to absorb some of the mixing water in the concrete mix, thereby reducing the specified water/cement ratio in the mix. For this reason, it was necessary to soak the recycled aggregate in water for some minutes and then air-dried them for 10 – 15 minutes before they were batched and mixed. In doing this, the pores in

the recycled aggregate that would have absorbed the concrete mixing water was filled up by moisture, so that the aggregate is left in a saturated surface dried condition and thus will not absorb the concrete mixing water. For the natural coarse aggregate, the moisture absorption was observed to be 0%, this indicated that the natural coarse aggregates were not absorbing any moisture and thus would have no need to be soaked in water before use. The recycled aggregate with moisture absorption of 4.6%, used in this work was ok as it met the limit of moisture absorption for recycled coarse aggregate of 3.7% - 8.7% according to [5].

Table 3 shows the batching mass of the HPC mixture components and the results of average slump for 46 batches of concrete, including two batches of reference mixes for reference purposes.

Table 4 is the final regression table after model reduction from the initial regression analysis. The coefficients in the table are the estimated model coefficients. Summary statistics like S, R², R² (Pred.), and R² (adj.), were used to examine the goodness of fit of the developed model. The relatively low value of S and high percentage of R² (adj.), indicate that the model is a good fit to the

$$y = -1966225*x_1 - 2132746*x_2 + 7087461*x_3 - 246436*x_4 + 248676*x_5 + 248799*x_6 + 446914*x_7 + 17330515(x_1*x_2) - 25587799(x_2*x_3) - 12692418(x_3*x_7) + 20164(x_4*x_5) \quad (6)$$

Where y is the Slump in mm and x₁ - x₇ are the volume fractions of the mixture components mentioned earlier.

The model developed could be used to optimize the proportions of mixture components of the HPC, to obtain an optimized response property desired (workability). However, the desired workability would have to be within the range of response property obtained from laboratory experiment in this work. The “response optimizer” function of MINITAB 17 was used to perform the optimization

analyzed data, and affirms the validity of the model to an extent.

Figure 1 shows the gradation curves of the natural and recycled coarse aggregates used in this work. The natural coarse aggregate appears to be well graded, and the recycled coarse aggregate, though not as well graded as the natural coarse aggregate, still has an acceptable range of gradation. The gradation curve of fine aggregate in Figure 2, on the other hand, shows an almost perfect gradation curve, indicating a well graded nature of fine aggregate.

A more thorough examination of the validity of the model was done using the residual plot in Figure 3. An examination of the residual plots confirmed the conformity of the developed models to the least square’s model assumptions on which the developed models are based. The normality condition was confirmed from the normal probability plot, the linearity condition and the equal variance condition were confirmed from the “residual versus fit plot”, and the final model is as given in equation (6).

procedure. To obtain a slump of say 155 mm the response optimizer function of MINITAB 17 predicted the following component proportions for use: x₁=0.161601, x₂=0.129118, x₃=0.015, x₄=0.0127065, x₅=0.199254, x₆=0.200908, x₇=0.281413. An experiment was carried out in the laboratory using the components proportions as predicted by MINITAB 17. A slump of 155 mm was observed from the experiment. This confirmed the high degree of accuracy of the developed model for the Slump of the HPC, which measures its workability.

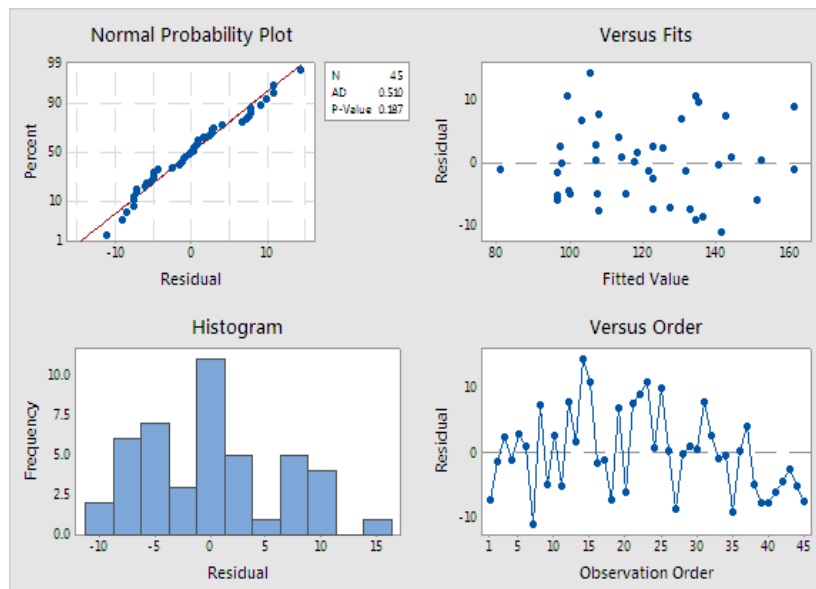


Figure 3: Residual plot for Slump

V. CONCLUSION AND RECOMMENDATION

In this work, a model was formulated for the workability of high performance recycled coarse aggregate concrete. The formulated model was used to optimize the workability property of the resulting concrete. This model is recommended for use in the determination of workability of high performance recycled coarse aggregate concrete. However, the range of desired workability should be within the range of workability obtained from laboratory experiments in this work, which is 80 mm – 170 mm for slump, and the mixture components should be of the same quality as those used in this work.

REFERENCES

- [1]. Mehta, P. K, Monteiro, J. M., Concrete, microstructure, properties, and materials. New York: McGraw Hill,2006.
- [2]. Neville, A. M. 2011. Properties of concrete. Edinburg: Pearson Education Limited.
- [3]. M. Malešev, V. Radonjanin, S. Marinković, Recycled Concrete as Aggregate for Structural Concrete Production, *Journal Sustainability* 2010, Vol. 2–05 (2010) 1204–1225.
- [4]. V. Radonjanin, M. Malešev, Recycled aggregate concrete - composition, properties and application, 2008, Vol. 40 (2007) 48–91.
- [5]. J. S. Ryou, and Y. S. Lee, "Characterization of Recycled Coarse Aggregate (RCA) via a Surface Coating Method" *International Journal of Concrete Structures and Materials*, 2014, Vol.8, No.2, pp 165-172.
- [6]. Cornell, J. A. (2011). *A premier on Experiment with Mixture*. New Jersey: John Wiley & Sons.
- [7]. Ettu, L. O., Arimanwa, J. I., Anya, U. C., and Effiong, E. E., " Optimization Of The Compressive Strength Of High Performance Recycled Coarse Aggregate Concrete." *IOSR Journal of Mechanical and Civil Engineering (IOSR-JMCE)*, 18(4), 2021, pp. 35-42.
- [8]. Minitab, Inc. Minitab Statistical Software, Release 17 for Windows, State College, Pennsylvania, 2014. Minitab® is a registered trademark of Minitab, Inc.
- [9]. Al Guru Fosroc LLC, Detailed information on High-range water reducing admixture (Conplast SP 430).
- [10]. British Standard Institution. BS 1377-2. Methods of test for Soils for civil engineering purposes — Part 2: Classification tests. London, 1990
- [11]. British Standard Institution. BS EN 1097-3. Test for Mechanical and Physical Properties of aggregates - Part 3. Determination of Loose Bulk density and voids. London, 1998.
- [12]. British Standard Institution. BS 812: Part 3. Testing aggregates Part 3: Methods for determination of mechanical properties. London, 1995
- [13]. Scheffe, H. (1958). Experiment with mixtures. *Journal of the royal statistical society*. 20, 344-360
- [14]. Simon M. J., Concrete Mixture optimization using Statistical Methods: Final Report. FHWA-RD-03-060, 2013.
- [15]. BS EN 12350-2. Testing fresh concrete. Method for Determination of Slump. British Standard Institute, 2000.
- [16]. British Standard Institution. BS EN 1097-2. Tests for mechanical and physical properties of aggregates Part 2. London 2020.