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Removal of Chromium from Enugu Coal using HCl as Leaching Agent

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ABSTRACT: This study investigated the removal of chromium from Enugu Coal with HCL as the leaching agent under different conditions such as Leaching Time, particle size, acid concentration, and volume of leaching agent. The filtrate from each treatment was analyzed with Atomic Absorption X-ray Spectrometer (AAS) to determine the amount of Chromium leached. Similarly, the residual coal from each treated sample was also analyzed together with virgin coal using Scanning Electron Microscopy (SEM). Micrograph of virgin coal revealed the features of lithhophiles like aluminum and silicates. The absence of these features on the residual coal confirmed the removal of inorganic elements in residual samples. A quadratic model was predicted and optimized which resulted to Particle size of 50µm, reaction time of 35 hours, HCL concentration of 2mol/dm^3 and Leaching agent of volume of 100mls. Optimum conditions were validated at model desirability of 1. Experimental value of 91.19% with error of 0.840% was removed.

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Energy has become an important prerequisite for the economic development of a country. On one hand it is used for the industrial and agricultural purposes and on other hand it is required for domestic use. Nigeria is presently facing an acute power shortage, with a rapidly growing population and economy, and relying heavily on thermal power generation and hydroelectric power. About half of the thermal power generation is based on oil or on natural gas. Oil is very expensive and rising unpredictable to unprecedented height even though Nigeria is one of the largest oil producers in the world. Nigeria is presently facing a high demand and supply of gas for electricity and may increase in the coming years despite federal government efforts to increase power generation before 2020.

One of the strategic issues for contemporary manhood is production of a sufficient amount of energy for further technological development. Despite numerous attempts to use new, practical inexhaustible energy sources, such as solar energy (Lowery, 2002), wind energy (Thoma, 1992), and high and low tides (Omer, 2002).On the basis of instigations of coal genesis, its composition, as well as general characteristics of coal deposits, coal can be defined as a combustible sedimentary rock, originating mainly (some coals are algal) from residues of terrestrial and aquatic plants, and of minerals (Wood et al, 1983). Coal can also be defined as a solid dark – colored, carbon – rich material that occurs in stratified sedimentary deposits. Coal happens to be the most abundant of all mineral resources in Nigeria.

Table 1: Nigerian coal fields and reserves

Location	Type	Deposits in Million Tones		
		Indicated	Inferred	Total
Anambra state				
Enugu	Sub-bituminous	29.5	17.3	46.8
Inyi	"	10.3	-	10.3
Umuahia/Belt	Lignite	UNDER	Investigation	
Nnewi	"	"	"	"
Benue State				
Orukpa	Sub-bituminous	50.9	70.1	130.0
Okaba	"	54.9	190.3	245.2
Ogboyoga	"	83.3	310.3	393.8
Idah	Sub-bituminous	UNDER	Investigation	
Koton Keffi	"	"	"	
Oturkpo	"	"	"	
Dekina	Coking	"	"	
Bauch state				
Gombe	Coking	"	"	
Plateau State				
Lagia	Coking	22.0	-	22.0
Lamja	"	UNDER	Investigation	
Bendel State				
Asaba	Lignite	"	"	
		293.6	708.4	989.0

Source: (Onwu, 1999): Coal fundamentals and conversion technology

Unfortunately it is the least tapped. Coal, if properly tapped and harnessed, could be a source of foreign exchange for the nation. Leaching of coal samples before combustion or before use for power plant operations will surely remove most of the trace elements in that coal thereby minimizing its environmental problems. Removal of materials by dissolving them away from solids is called leaching. Enugu coal has been found to contain greater quantity of chromium and lead when compared with other trace elements according to (Onwu, 1999). Therefore, this effort is aimed at optimizing the removal (leaching) of chromium from coal sample using response surface methodology.

MATERIALS AND METHOD

Raw materials and its source: The main raw materials used in this research included Bituminous coal, hydrochloric acid and distilled water. The coal sample was obtained from Onyeama mine Enugu through Enugu Coal Co-operation while the mineral acid and distilled water was purchased from Ogbete main market Enugu Nigeria.

Coal sample Preparation: The coal sample obtained was first wet washed with distilled water to remove free sand particles associated with the coal and dried under the sun for 24hours. Cleaned coal sample was crushed followed by grinding in a pestle and mortar, screened through 75µM sieves using a sieve shaker. The definite sized coal sample was dried in a vacuum oven at 110°C for one hour and cooled in a desicator.

Characterization of coal sample: Moisture content, Ash content, volatile matter and Fixed Carbon were analyzed by loss on ignition using the standard method (ASTM D2974-14, 2014). An FS 240 variant Atomic Absorption Spectrophotometer (AAS) using Nitrous oxide oxidant gas, Acetylene gas, Air oxidant gas, distilled water, and conical flask were used for the analysis of chromium. The micrograph of the virgin and residual coal samples was determined using Scanning Electron Microscope (SEM).

Extraction of trace metal (Chromium): 5grams of pulverized coal sample was measured into a 100ml beaker of different volumes of HCL of different concentrations. The mixture was shaken vigorously and placed in a water bath set at 65°C for 20hours with intermittent stirring. The mixture was filtered using what man filter paper No. 1. The filtrate was then taken for analysis while the residue was washed thoroughly with several amount of distilled water to remove all traces of acid. The washed coal residue was placed in the oven set at 50°C to dry and weighed until constant weight was obtained. The dried coal residues were placed in a desiccator to cool. The washed coal residue was taken to the laboratory for further analysis.

Experimental plan using Central Composite Design (CCD): The Central Composite Design (CCD) was used to study the effects of the variables towards their responses and subsequently in the optimization studies. This method is suitable for fitting a quadratic surface and it helps to optimize the effective parameters with a minimum number of experiments, as well as to analyze the interaction between the parameters. In order to describe the effects of Particle size, acid concentration, Process duration, and leachant volume on the percentage removal of trace metals from coal samples, batch experiments were conducted based on the design. The coded values of the process parameters were determined by equation 1, (Chen *et al,* 2011)

$$x_i = \frac{X_i - X_o}{\Delta X} \quad (1)$$

Where x_i – coded value of the ith variable, X_i – uncoded value of the ith test variable and X_o – uncoded value of the ith test variable at center point

Table 2: Factor Levels of Independent Variables for leaching of Trace Element (Chromium) from Enugu Coal

Independent Factors	-α	Low level(-)	Medium level(0)	High level(+)	+α
Particle Sizes (µm)	45	50	55	60	65
Time (hours)	20	25	30	35	40
Acid Conc (mol/dm^3)	0.5	1.0	1.5	2.0	2.5
Leachant Volume (mls)	85	90	95	100	105

RESULTS AND DISCUSSION

Results of the proximate analysis of Enugu coal: Proximate analysis of the virgin coal sample with particle size of 75µM was conducted and results presented as shown in table 4. It was observed from table 4 that the total moisture content, volatile matter content, ash content and fixed carbon obtained are 5.1%, 23.6%, 7.4% and 39.2% respectively and these values very much in agreement with the results reported by other researchers such as Onwu, 1990.From the results in table 4, it can be concluded that the coal sample studied is sub-bituminous coal according to American society for testing and materials (ASTM) classification of coal.

Table 3: Central Composite Design of experiment in terms of real values

STANDARD	RUN	FACTOR 1 Particle Size (µm)	FACTOR 2 Time (hours)	FACTOR 3 Acid Conc (mol/dm^3)	FACTOR 4 Leachant Volume (mls)
12	1	60.00	35.00	1.00	100.00
23	2	55.00	30.00	1.50	85.00
14	3	60.00	25.00	2.00	100.00
30	4	55.00	30.00	1.50	95.00
27	5	55.00	30.00	1.50	95.00
28	6	55.00	30.00	1.50	95.00
21	7	55.00	30.00	0.50	95.00
18	8	65.00	30.00	1.50	95.00
26	9	55.00	30.00	1.50	95.00
17	10	45.00	30.00	1.50	95.00
22	11	55.00	30.00	2.50	95.00
5	12	50.00	25.00	2.00	90.00
1	13	50.00	25.00	1.00	90.00
7	14	50.00	35.00	2.00	90.00
6	15	60.00	25.00	2.00	90.00
9	16	50.00	25.00	1.00	100.00
8	17	60.00	35.00	2.00	90.00
15	18	50.00	35.00	2.00	100.00
25	19	55.00	30.00	1.50	95.00
4	20	60.00	35.00	1.00	90.00
24	21	55.00	30.00	1.50	105.00
2	22	60.00	25.00	1.00	90.00
13	23	50.00	25.00	2.00	100.00
16	24	60.00	35.00	2.00	100.00
10	25	60.00	25.00	1.00	100.00
20	26	55.00	40.00	1.50	95.00
3	27	50.00	35.00	1.00	90.00
11	28	50.00	35.00	1.00	100.00
29	29	55.00	30.00	1.50	95.00
19	30	55.00	20.00	1.50	95.00

Table 4: Proximate analysis of virgin coal sample

Characteristic properties	**Percentage weight %**
Surface moisture	0.90
Inherent moisture	4.20
Total moisture content	5.10
Volatile matter	23.60
Ash content	7.40
Fixed carbon	63.90

*SEM Observations***:** SEM micrographs of the virgin and leached coal samples are provided in figs.1 –2. Figs.1a-1b represents the SEM image of the virgin coal at 10µm and 30µm. It can be observed from the figure that a bulk of microstructure which in turn is composed of a homogeneously distributed network comprised of small filamentous and fistulous crystallites showing the presence of minerals. In the matrix, Luminous and non-luminous features can be seen. These features indicate the presence of minerals distributed in the organic matrix and as surface coverage. Some features such as fissures, cleats, cracks and veins can also be seen. To remove the minerals and enrich the coal in usable carbon, chemical leaching was performed with HCL acid and the mineralogical studies of the residual coal performed. From figs 2a and 2b, it can be seen that numerous aggregated particles was present, the porosity has been increased and provides strong evidence that significant amounts of inorganic elements are being removed. Also from figs. 2a and 2b, it was observed that the process caused serious morphological changes in the particles and did enormous harm to the surface by leaching some of the inorganic elements.

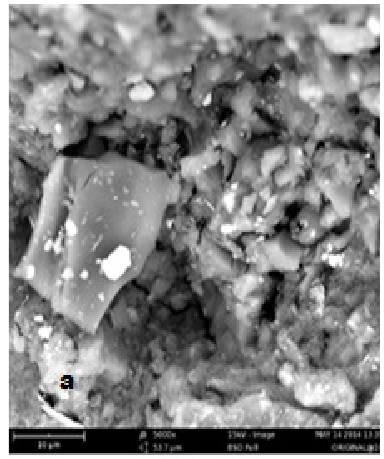

Fig 1a: SEM Micrograph for Virgin Coal at 10µM

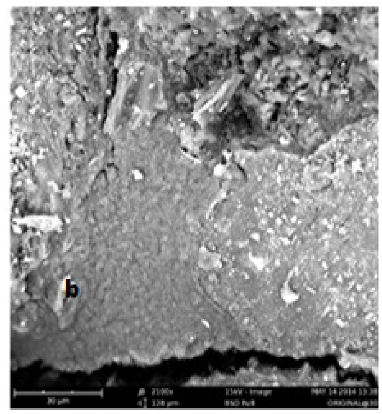

Fig 1b: SEM Micrograph for Virgin Coal at 30µM

However the leachants used with the combination of factors for this experiment seems to be more effective in removing trace elements from the coal under study.

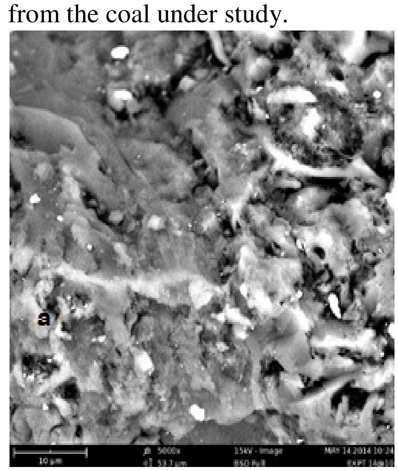

Fig 2a SEM Micrograph of residual coal (Treated) at 10µM

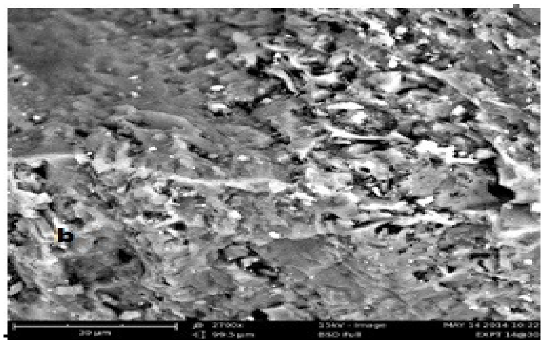

Fig 2b SEM Micrograph of residual coal(Treated) at 30µM

Mineral Analysis of Coal Samples: The results of the mineral analysis of the filtrate based on the experimental design of table 3 were shown in table 5. The trace metal analyzed was chromium. This element was mentioned by prior researchers (Alafara*et al,* 2009), as one of the elements that contribute to environmental problems during coal combustion. Design Expert 8.0.7.1 trial version was employed for the analysis of the results as shown in table 5. The summary of P-values indicates that a quadratic model fitted the ANOVA analysis and hence it was suggested. The linear and 2FI models were not suggested. The Cubic model is always aliased because the Central Composite Design does not contain enough runs to support a full cubic model. From table 6, a significance level of 5% was used hence all terms whose P-value are less than 0.05 are considered significant therefore X_1, X_2, X_3, X_4, X_1X_4, X_2X_4, X_3X_4, X_1^2, X_2^2, X_3^2, and X_4^2 are significant. The regression F-values of 9.499E+005 implies that the model is significant which was validated by the P-values being less than 0.0001. The tests for adequacy of the regression models, significance of individual of model coefficients and the lack of fit test were performed using the same statistical package. The P values were used as a tool to check the significance of each of the coefficients, which in turn are necessary to understand the pattern of the mutual interactions between the test variables (Shrivastava *et al,* 2008). The larger the magnitude of F-test value and the smaller the magnitude of P-values, the higher the significance of the corresponding coefficient (Josh et al, 2014). The adjusted R^2 (0.9265) is in close agreement with the predicted R^2 (0.7683).

Table 5: Central Composite Design of experiment with results of the percentage chromium leached from Enugu coal

std	Run	Factor 1 X_1: Particle size (µm)	Factor 2 X_2:Time (hours)	Factor 3 X_3:Acid Conc (mol/dm^3)	Factor 4 X_4:Leachate volume (mls)	Response 1 Percentage Chromium leached
12	1	60.00	35.00	1.00	100.0	87.00
23	2	55.00	30.00	1.50	85.00	73.63
14	3	60.00	25.00	2.00	100.0	78.58
30	4	55.00	30.00	1.50	95.00	77.28
27	5	55.00	30.00	1.50	95.00	77.28
28	6	55.00	30.00	1.50	95.00	77.28
21	7	55.00	30.00	0.50	95.00	80.92
18	8	65.00	30.00	1.50	95.00	81.44
26	9	55.00	30.00	1.50	95.00	77.28
17	10	45.00	30.00	1.50	95.00	90.27
22	11	55.00	30.00	2.50	95.00	79.26
5	12	50.00	25.00	2.00	90.00	76.97
1	13	50.00	25.00	1.00	90.00	85.82
7	14	50.00	35.00	2.00	90.00	86.16
6	15	60.00	25.00	2.00	90.00	80.56
9	16	50.00	35.00	1.00	100.0	88.85
8	17	60.00	35.00	2.00	90.00	82.13
15	18	50.00	35.00	2.00	100.0	91.19
25	19	55.00	30.00	1.50	95.00	77.28
4	20	60.00	25.00	1.00	90.00	76.57
24	21	55.00	30.00	1.50	105.0	75.05
2	22	60.00	25.00	1.00	90.00	87.65
13	23	50.00	25.00	2.00	100.0	81.62
16	24	60.00	35.00	2.00	100.0	80.53
10	25	60.00	25.00	1.00	100.0	84.04
20	26	55.00	40.00	1.50	95.00	89.48
3	27	50.00	35.00	1.00	90.00	82.48
11	28	50.00	35.00	1.00	100.0	85.75
29	29	55.00	30.00	1.50	95.00	77.28
19	30	55.00	20.00	1.50	95.00	88.40

Table 6: ANOVA analysis for leaching of Chromium

Source	Sum of square	df	Mean square	F value	p-value Prob>F
Model	820.11	14	58.58	9.499E+005	<0.0001
X_1-particle size	116.82	1	116.82	1.894E+006	<0.0001
X_2-Time	3.49	1	3.49	56569.26	<0.0001
X_3-Acid conc	4.09	1	4.09	66356.82	<0.0001
X_4-leachant vol	2.99	1	2.99	48473.58	<0.0001
X_1X_2	58.18	1	58.18	9.434E+005	<0.0001
X_1X_3	3.14	1	3.14	50947E+005	<0.0001
X_1X_4	44.12	1	44.12	7.155E+006	<0.0001
X_2X_3	159.96	1	159.96	2.594E+006	<0.0001
X_2X_4	0.15	1	0.15	2372.53	<0.0001
X_3X_4	2.63	1	2.63	42689.29	<0.0001
X_1^2	126.11	1	126.11	2.045E+006	<0.0001
X_2^2	288.03	1	288.03	4.671E+006	<0.0001
X_3^2	13.56	1	13.56	2.198E+005	<0.0001
X_4^2	14.80	1	14.80	2.399E+005	<0.0001
Residual	9.250E-004	15	6.167E-005	-	-
Lack of fit	9.250E-004	10	9.250E-005	-	-
Pure error	0.000	5	0.000	-	-
Cor Total	820.11	29	-	-	-

Std. Dev. = 2.04; Mean = 91.23; C.V. = 2.23%; PRESS = 378.76
R-Squared = 0.9620 Adj R-Sq = 0.9265; Pred R-Sq = 0.7683; Adeq Precision = 19.109

The adequate precision measures the signal to noise ratio and compares the range of the predicted value at the design points to the average prediction error. The adquate predicion ratio above 4 indicates adequate model efficacy . Hence, the adequate precision ratios of 19.109 indicates adquate model efficacy. Also, a PRESS value of 378.76 indicates an adequate signal implying that the models can be used to navigate the design space. The coefficient of regression R^2 was used to validate the fitness of the model equation. The R^2 has a high value of 0.9620 showing that 96.20% of the variability in the response can be explained by the model. This implies that the prediction of experimental data is quite satisfactory. The quadratic model equations obtained for the leaching are presented as shown in equation 2 and 3

Regression Equation in Terms of Coded Factors::
Amount of Chromium Leached (%) = +77.28-2.21* X_1-0.38 * X_2-0.41* X_3+0.35* X_4-1.91* X_1* X_2+0.44* X_1* X_3-1.66 * X_1* X_4+3.16 * X_2* X_3+0.096* X_2* X_4+0.41* X_3 *X_4 +2.14* X_1^2+3.24* X_2^2+0.70* X_3^2-0.73* X_4^2 (2)

Regression Equation in Terms of Actual Factors:
Amount of Chromium Leached =-151.70031-1.54329 Particle size-5.91888 * Time-72.36708 * Acid Conc+8.94787 * Leachant Vol-0.076275* Particle size* Time+0.17725* Particle size * Acid Conc-0.066425* Particle size * Leachant Vol+1.26475* Time* Acid Conc+3.82500E-003* Time* Leachant Vol+0.16225* Acid Conc * Leachant Vol+0.085771* Particle size^2+0.12962* Time^2+2.81208 * Acid Conc^2-0.029379 * Leachant Vol^2* (3)

In a regression equation, when an independent variable has a positive sign, it means that an increase in the variable will cause an increase in the response while a negative sign will result in a decrease in the response. A combination of the actual experimental response and the predicted response from the mathematical equations are given in table 7, where it was seen that there is a close correlation between the actual experimental response and the predicted response. This comfirms the effectiveness of the leaching of trace metals with conc HCL.

Model Validation of the leaching of trace metals: The optimum conditions predicted for the 92.03% for leaching of trace metals (Chromium) as given in the table 7 were as follows: Particle size 50µm; time, 35 hours; Acid Conc 2.0 M and Leachant Volume 100mls. This value is in close agreement with the experimental value of 91.19% performed at the same optimum values of the process variables.

The Normal plot of Residuals as shown in fig 3 and the Predicted vs Actual plots as shown in fig 4 were used to check whether the points will follow a straight line in which we conclude that the residuals follow a normal distribution.

Table 7: Actual and Predicted values for the leaching of Trace Element (Chromium) from Enugu Coal

std	Run	Factor 1 X_1: Particle size (µm)	Factor 2 X_2:Time (hours)	Factor 3 X_3:Acid Conc (mol/dm^3)	Factor 4 X_4:Leachate volume (mls)	Experimental Value (%)	Predicted Value (%)
12	1	60.00	35.00	1.00	100.0	87.00	73.33
23	2	55.00	30.00	1.50	85.00	73.63	73.64
14	3	60.00	25.00	2.00	100.0	78.58	78.59
30	4	55.00	30.00	1.50	95.00	77.28	73.31
27	5	55.00	30.00	1.50	95.00	77.28	75.91
28	6	55.00	30.00	1.50	95.00	77.28	74.01
21	7	55.00	30.00	0.50	95.00	80.92	81.12
18	8	65.00	30.00	1.50	95.00	81.44	88.40
26	9	55.00	30.00	1.50	95.00	77.28	75.51
17	10	45.00	30.00	1.50	95.00	90.27	90.10
22	11	55.00	30.00	2.50	95.00	79.26	76.44
5	12	50.00	25.00	2.00	90.00	76.97	85.52
1	13	50.00	25.00	1.00	90.00	85.82	86.12
7	14	50.00	35.00	2.00	90.00	86.16	90.08
6	15	60.00	25.00	2.00	90.00	80.56	82.13
9	16	50.00	35.00	1.00	100.0	88.85	90.11
8	17	60.00	35.00	2.00	90.00	82.13	79.50
15	18	50.00	35.00	2.00	100.0	91.19	92.03
25	19	55.00	30.00	1.50	95.00	77.28	78.14
4	20	60.00	25.00	1.00	90.00	76.57	83.30
24	21	55.00	30.00	1.50	105.0	75.05	79.31
2	22	60.00	25.00	1.00	90.00	87.65	86.72
13	23	50.00	25.00	2.00	100.0	81.62	89.91
16	24	60.00	35.00	2.00	100.0	80.53	90.91
10	25	60.00	25.00	1.00	100.0	84.04	89.07
20	26	55.00	40.00	1.50	95.00	89.48	89.10
3	27	50.00	35.00	1.00	90.00	82.48	88.91
11	28	50.00	35.00	1.00	100.0	85.75	87.55
29	29	55.00	30.00	1.50	95.00	77.28	78.60
19	30	55.00	20.00	1.50	95.00	88.40	88.33

Table 8: Numerial optimization for the leaching of chromium from Enugu coal using HCL

Model desirability	Particle size (µm)	Time (hours)	Acid conc (mole/dm^3)	Leachant volume (mls)	Leaching Capacity %		
					Predicted	Experimental	% Error
1.0	50	35	2.0	100	92.03	91.19	0.84

Hence, it can be seen from the figures that the points were closely distributed to the striaght line of the plot, it confirms the good relationship between the experimental values and the predicted values of the response though some small scatter like an “S” shape is always expected. These plots equally confirm that the selected model was adequate in predicting the response variables in the experimental values.

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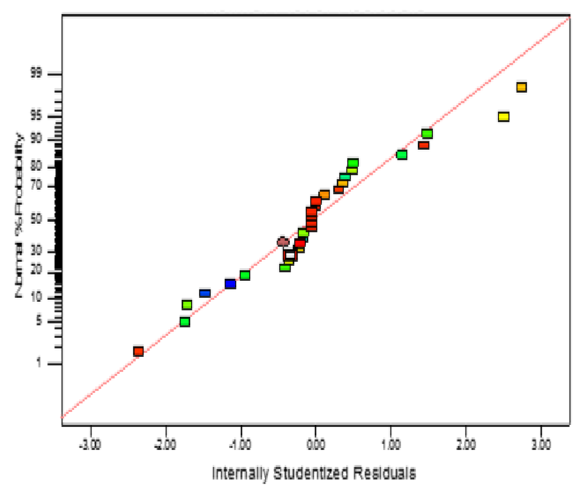

Fig 3: Normal Plot of Residuals for the leaching of trace element (chromium)

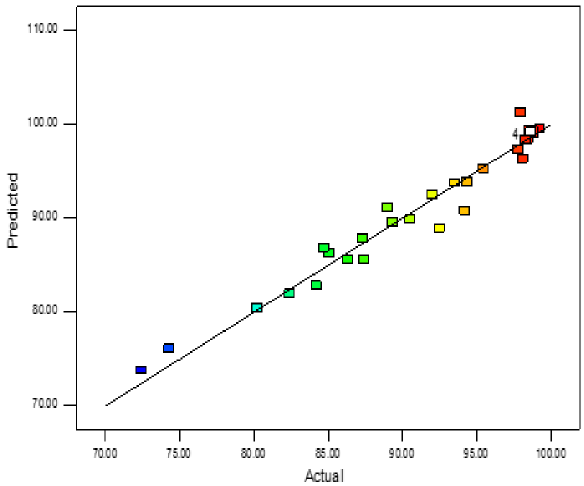

Fig 4: Predicted Vs Actual plot the leaching of trace metal (chromium)

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