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Survey on solid wastes management by composting: Optimization of key process parameters for biofertilizer synthesis from agro wastes using response surface methodology (RSM)

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ABSTRACT

The optimization of key process parameters for the transformation of agro wastes into biofertilizer has been demonstrated using response surface methodology (RSM). Biofertilizer was produced by composting using 120 L capacity drum made of polyethylene as the composter. Composting time (X_1), dosage ratio (X_2) and moisture content (X_3) were the independent factors while percentage nitrogen, phosphorus and potassium (N.P.K) were the response factors. The outcomes exhibited that composting time, dosage ratio and moisture content all significantly affects the mineralization of N.P.K at probability value of 0.0001. The coefficients of determination also called regression coefficients of 98.60%, 99.79% and 97.80% for nitrogen, phosphorus and potassium observed between the predicted and the real value are obvious that the developed regression models can fit the experimental data well. It was seen from the optimization studies that the pinnacle value of N.P.K from the ideal conditions are 9.62%, 8.97% and 5.62. Characterization of the composite uncovered that biofertilizer produced has a high potential for commercial application on agricultural land. It can be concluded that combination of sawdust, sewage sludge and vegetable waste is a good mixture for biofertilizer synthesis. Also, the nutrients release by the compost materials during the process of composting may be maximized when process conditions are circumspectly managed within the reported optimal value.

1. Introduction

In any emerging or modern society, waste management and control is dependably a characteristic piece of such society. Wellsprings of residential and commercial wastes have developed widely in Nigeria over the previous decade. The by-product of agricultural exercises is generally alluded to as "Agricultural Wastes" (Westerman and Bicudo, 2005). These wastes essentially appear as harvest deposits (leftover stalks, straw, leaves, roots, husks, shells etcetera) and animal waste (excrement). Agricultural wastes can be overseen by changing over to alternative manures (bio-fertilizer) through proper composting (Oltjen and Beckett, 2006). The management of solid wastes has dependably been a major issue to most urban areas in Nigeria. Aside Pyrolysis, burning in incinerator and storage, a large portion of the natural wastes from human, creature, rural and mechanical foundations presenting genuine ecological and medical issues can be overseen by anaerobic/aerobic fermentation producing biogas and biofertilizer individually through proper composting. These procedures are exceptionally worthwhile remembering that, they limit harm to the earth and create monetarily significant items from wastes (Ogazi and Omueti, 2000; stentiford, 1996). As indicated by (Stentiford, 1996; Dumitrescu et al., 2009), Composting is defined as biological oxidative degradation of organic matter in wastes under controlled conditions which allows the growth of micro-organisms that convert biodegradable natural wastes into an end product that is adequately stable for application in agricultural land without antagonistic ecological impacts. The end product of composting has been observed to be more valuable to plants and soil biodiversity Rasapoor et al.,(2009). Carbon dioxide, water, mineral particles and humus are the primary products of aerobic composting. The procedure decimates pathogens due the accelerated temperature by microbial exercises. Likewise, nitrogen fixing bacteria converts nitrogen from

Abbreviations: RSM, response surface methodology; CCD, central composite design; N, nitrogen; P, phosphorus; K, potassium; ASTM, American Society for Testing and Materials; ANOVA, analysis of variance; CV, coefficient of variation; TOC, total organic carbon; R^2, coefficient of regression or coefficient of determination.; AAS, atomic absorption spectrometer.

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unstable ammonia to stable natural structures. The procedure for the most part decreases the volume of waste. For the reason that composting is an effective strategy for reusing waste, this investigation has an extraordinary importance for most developed and developing nation that needs to deal with, consistently, bigger amounts of biodegradable waste, for example, a waste administration organization (Enugu State Waste Management Agency) in Enugu State Nigeria oversaw both as local waste (86,883 tons in 2010) and sewage sludge (53,750 tons in 2010). Sludge rich in natural and mineral mixes, for example, (nitrogen and phosphorus) and in lipids is created by treating modern effluents. At the point when sludge is arranged without treatment, it turns into a wellspring of contamination (Ilegbune, 2006). With the end goal to avert genuine natural issues, for example, sullying of groundwater by leachate, contamination of the air with foul gases and so forth, Sludge must be stabilized by chemical, organic and biochemical methods (Mowoe, 2001; Jimoh, 2005).

Optimization is a strong decision making which is the act of producing the best results under certain conditions (Datta, 2011). The main components of optimization are; (i) The objective function, (ii) The variables and (iii) The constraints. Optimization problems can be stated to maximize or minimize that is subject to the constraints. Optimization problems could be constrained optimization problems which are subject to constraints or unconstrained optimization problems which is subject to no constraints considering the nature of the equations for the objective functions and constrains. Optimization problems can be linear, non linear, geometric and quadratic programming problems.

Essential of process streamlining of key process parameters involved in the conversion of agro wastes cannot be overemphasized. The reason for statistically designing an experiment is to gather regular connection between different components influencing the procedure towards finding the most appropriate conditions. Process optimization was done in this work using response surface methodology (RSM). It is a statistical tool used mainly for optimization. RSM is for the most part utilized to study about the impact of independent factors on the response(s). It is additionally used to ponder the impacts of multiple variables and their cooperation. These different elements are the independent factors while the response(s) are the dependent variables (Datta, 2011). RSM relates item properties by utilizing regression equation that portrays interrelations between information factors and item properties (Adeyanju et al., 2016). The most prevalent and regularly utilized type of RSM is the central composite design (CCD) and Box Behnken design. In this work, central composite design was utilized for the process optimization. These designs are rotatable or near rotatable. The factors studied were composting time, moisture content (water solid ratio) and dosage ratio. Therefore, this research aims at contributing to waste management by converting some agro waste into biofertilizer through composting and afterward, optimize the key process parameters in composting using response surface methodology.

2. Materials and method

2.1. Materials

Raw materials used in this research were; sawdust, dewatered sewage sludge and vegetable wastes (comprising of different left over of fresh green leaves, watermelon, cabbage, lettuce, cucumber etc). The sawdust was sourced from local sawmill, the vegetable wastes was sourced from the local market and were shredded and homogenized with a cutter to improve decomposition during composting. Dewatered sewage sludge was sourced from waste water treatment plant of Wilson Industry Nigeria Limited Nsukka.

2.2. Design of composting drums

The composting was done in polyethylene drums of 120 L capacity. The drums were reasonably changed for air dissemination. The drums were changed by giving 10 mm equidistant gaps in six layers on the periphery of the drums utilizing a hand driller to encourage the air dissemination inside the drums. Two inspecting windows (one each at center and base part) were given in the drums to gather the intermittent samples for investigation. The arrangements for the leachate collection from the base of the drums were likewise given.

2.3. Composting operations

The composting process was carried out in open space to allow the natural aeration. The drum was supported on the bricks and the plastic tray was kept below the drum for the collection of leachate. For the study, 90 kg of the homogenized wastes samples at different dosage ratios as shown in Table 3 were added into the drums. Aeration was achieved by manual turning of the composts once daily. Various operating and product quality parameters such as pH, temperature, organic matter and total carbon were monitored as the compost last. Samples were taken from the drum after every 5 days for laboratory analysis. The moisture content was monitored and maintained using electronic moisture meter (Reotemp 648(800) San Diego CA).

2.4. Physiochemical characterization of the samples

The ASTM D2974-07 was used in the analysis of the percentage composition of organic matter, ash content and moisture content (ASTM, 2007). The nitrogen content was estimated by Kjeldahl's method Sobiecka et al. (2007). Phosphorus and potassium contents were analyzed using Atomic Absorption spectrophotometer (AAS) (Model 2010, VGP manufacturer USA). pH was measured in the filtrate solution using pH-meter 340I/SET (Texcare Instrument, New Delhi India, Precision /sensitivity 0.01/−59.16 mV/pH @25°C). Method for the determination of total organic carbon (TOC) in soil and sediment was used for organic carbon content determination (Schumacher, 2003).

2.5. Statistical analysis and mathematical modeling

In order to examine if there is a relationship between the dependent and independent variables, the data gathered were subjected to regression analysis utilizing response surface methodology of Design expert version 8.0.7.1. Regression analysis was utilized to show a response (Y_i) as a scientific capacity of a couple of consistent elements. Every response (Y_i) was represented by mathematical equation that relates the response surfaces. The response was represented as second-order polynomial equation as indicated by Eq. (1).

$$Y_i = f(y) = \beta_0 + \sum_{i=1}^{k} \beta_i X_i + \sum_{i=1}^{k} \beta_{ii} X_i^2 + \sum_{i=1}^{k} \sum_{i=1}^{k} \beta_{ij} X_i X_j + \varepsilon \quad (1)$$

where Y_i is the predicted response used to relate the independent variables, k is the number of independent variables$X_i (i = 1,2,3)$; while β is a constant coefficient and β_i, β_{ij} and $\beta_{ii}$is the linear, interaction and square terms respectively and ε is the random error term. Multivariant regression analysis with model Eq. (1) was carried out on data using design expert 8.0.7.1 software to yield Eq. (2) which was used to optimize

Table 1
Coded and un-coded values of the independent factors.

Code	Actual value of independent variable
$-\alpha$	X_{min}
-1	$\frac{(\alpha-1)X_{max} + (\alpha+1)X_{min}}{2}$
0	$\frac{X_{max} + X_{min}}{2}$
$+1$	$\frac{(\alpha-1)X_{min} + (\alpha+1)X_{max}}{2}$

Table 2
Factor levels of independent variables for the synthesis of biofertilizer.

Independent factors	−α (Axial)	Low level	Medium level	High level	+α (Axial)
	−1.68	−1	0	+1	+ 1.68
Process duration X_1	21.59	25	30	35	38.41
Dosage ratio X_2	2.32 (1:2:1)	3 (1:1:1)	4 (2:1:1)	5 (2:2:1)	5.68 (3:1:1)
Moisture content X_3	46.59	50	55	60	63.41

the product responses.

$$Y = \beta_0 + \beta_1 X_1 + \beta_2 X_2 + \beta_3 X_3 + \beta_{11} X_1^2 + \beta_{22} X_2^2 + \beta_{33} X_3^2 + \beta_{12} X_1 X_2 + \beta_{13} X_1 X_3 + \beta_{23} X_2 X_3 + \varepsilon \quad (2)$$

The model developed for every determination was then inspected for significance and lack of fit, while response surface plot was structured after removal of the non-significance terms with the same software. RSM was utilized in enhancing the procedure parameters for composting. The composting times (X_1), dose ratio (X_2) and moisture content (X_3) were the independent variables studied to optimize the yield (Y) of nitrogen, phosphorus and potassium. The independent factors were coded to lie at ±1 for the factorial point, 0 for the center point and ± <alpha> for the axial points as demonstrated in Table 1. The factors were signified by Eq. (3);

$$Z_j = \frac{X_i - X_j}{\Delta_j}; i = 1, 2, 3 \quad (3)$$

where Z_j are the coded values of the independent variables; while X_i and X_j are its real values and real value at the central point respectively. Δ_j is the step change of the variable X_i. The coded levels of the independent variables used in the RSM design were as shown in Table 2.

2.6. Statistical design and data interpretation

The central composite design (CCD) was utilized to contemplate the impacts of the factors towards their responses and subsequently in optimization studies. This technique is appropriate for fitting a quadratic surface and it improves the viable parameters with a minimal number of experiments, and in addition to investigate the association between the parameters. In order to describe the impacts of composting time, dosage ratio, and moisture content on the synthesis of biofertilizer, batch experiment were performed which was dependent on the CCD. In order to define the experimental range, preliminary experiment were first performed. As the structure ranges were built up, they were coded to lie at ±1α for the factorial point, 0 for the center point and ± 1α for the axial points. The codes were ascertained as an element of the scope of enthusiasm of each factor as appeared in Table 1. In this investigation, a small scale composting was conducted utilizing a mixture of sawdust, dewatered sewage sludge and vegetable wastes to create biofertilizer. The plan depended on five dimensions of the three factors as appeared in Table 2. The independent factors considered were composting time (25-35 days), dosage ratio (3–5 w:w) and moisture content (50–60%). Every single other parameter were kept constant. The working extents and five institutionalized dimensions were built up after a few fundamental runs. In light of CCD, the test runs contain 20 trials (8 factorial points, 6 center points and 6 axial points). Every one of the treatment were perfumed in randomized order. RSM and second order of three factors (composting time (X_1), dosage ratio (X_2) and moisture content (X_3), five level combination coded as −1.68, −1, 0, +1, and +1.68 as demonstrated by (Nahemiah et al., 2015; Snedecor and Cochran, 2008), was adopted to decide the impacts of the independent factors on the responses (N.P. K). Utilizing the coded dimensions, the typical dimensions were computed and delineated in Table 4, including 20 trial runs and diverse formulation compositions. The independent factors having the least P-value (or the most noteworthy F-ratio) demonstrates the most critical ($P < 0.05$) impact on the reliant factors (Samaram et al., 2015; Yolmeh et al., 2014). The non-significant terms ($P > 0.05$) were expelled from the created model in this investigation, with the exception of in a circumstance whereby a quadratic or collaboration impact including that factor would be critical (Samaram et al., 2015). Examination on the productivity of the model was finished by the assurance of the number of significant terms, regression equation P-value, lack of fit P-value and coefficient of regression (R^2) Yolmeh et al., 2014.

The outcome with R^2 values near 1 demonstrates that the model is more exact. The high value of adjusted and predicted coefficient of determination is additionally a sign of the sufficiency of the model fits for the experimental data, Yolmeh et al. (2014). The process optimization was done utilizing graphical and numerical advancement methods to decide the optimum composting conditions. The interaction impact was likewise considered utilizing the three dimensional (3D) surface plots obtained from the final model (Samaram et al., 2015). The yield of N.P.K would be compared with the predicted response values got from the final reduced model in order to demonstrate both the viability

Table 3
Physiochemical properties of the raw materials before composting.

Parameters	Organic waste		
	Sawdust	Vegetable waste	Sewage sludge
Moisture content (%)	23.3±6.5	37.8±6.5	33.6±6.5
Total Organic carbon (%)	58.2±0.5	23.6±0.5	24.2±0.5
Ash content (%)	24.3±0.1	17.8±0.1	22.8±0.1
Nitrogen (N) (%)	0.9±0.1	4.34±0.1	3.36±0.1
Phosphorus (P) (%)	1.04±0.1	6.4±0.1	3.2±0.1
Potassium (K) (%)	0.6±0.1	4.7±0.1	1.89±0.1
pH	5.87±1.0	7.15±1.0	7.23±1.0
Organic matter (%)	65±1.0	77±1.0	86.4±1.0

Table 4
Real value of independent factors with responses.

Std	Run	Factor 1 X_1: Time (days)	Factor 2 X_2: Dosage ratio (w: w)	Factor 3 X_3: Moisture content (%)	Response 1 Nitrogen (%)	Response 2 Phosphorus (%)	Response3 Potassium (%)
9	1	21.59	4.00	55.00	9.62	8.01	4.64
3	2	25.00	5.00	50.00	9.45	8.27	5.08
14	3	30.00	4.00	63.41	8.61	7.28	4.49
1	4	25.00	3.00	50.00	9.01	7.43	3.95
17	5	30.00	4.00	55.00	9.31	8.63	4.45
12	6	30.00	5.68	55.00	9.21	7.87	5.62
5	7	25.00	3.00	60.00	9.24	8.05	5.41
16	8	30.00	4.00	55.00	9.31	8.61	4.45
6	9	35.00	3.00	60.00	9.38	8.97	5.13
4	10	35.00	5.00	50.00	8.23	7.08	4.97
20	11	30.00	4.00	55.00	9.31	8.62	4.45
2	12	35.00	3.00	50.00	8.74	7.73	4.54
13	13	30.00	4.00	46.59	8.03	6.91	4.18
11	14	30.00	2.32	55.00	9.42	8.9	5.27
19	15	30.00	4.00	55.00	9.31	8.64	4.45
10	16	38.41	4.00	55.00	8.72	7.78	4.36
15	17	30.00	4.00	55.00	9.31	8.7	4.45
18	18	30.00	4.00	55.00	9.31	8.63	4.45
7	19	25.00	5.00	60.00	9.03	7.49	5.37
8	20	35.00	5.00	60.00	8.6	6.7	4.47

and unwavering quality of the regression fitted for the expected responses (Samaram et al., 2015).

3. Results and discussion

3.1. Nutrient composition in the raw materials

Percentage composition of nitrogen in sawdust as shown in Table 3 showed that sawdust must be blended with other nitrogen rich organic wastes before composting to maintain some level of nitrogen in the compost which would sustain the organisms responsible for biodegradation. Sewage sludge and vegetable wastes contained reasonable percentage nitrogen 4.34% and 3.36% as shown in Table 3. The values agree closely with the work done by Okon (2000), which reported that the value of nitrogen in dewatered sewage sludge and vegetable wastes before aerobic composting are 3.85% and 4.79%. These results showed that these wastes can supply the amount of nitrogen which can initiate the growth of microbes and enhance biodegradation. The organic matter was observed to be high in all the raw materials as presented in Table 3. The results agree with the report by Gajalakshmi and Abbasi (2008); which states that high percentage of organic matter in waste samples is an indication that they are good substrates for bio-fertilizer production.

3.2. Experimental design and model formulations

Table 2 shows the experimental parameters, ranges and level of independent variables examined in this work and the results are shown in Table 4. As regards to regression analysis, model fitting is the process of developing a probabilistic model that best describes the relationships between the dependent and independent variables. RSM was applied in developing the model and optimization of the process by first performing series of experimental runs (Table 4) to adequately and reliably measure the variables response before developing mathematical model of second order response surface best fit, and finally determine the optimal set of experimental parameters producing the optimal response value Damirel and Kayan (2012). In this study, effect of composting time (X_1), dosage ratio (X_2), and moisture content (X_3) and their interactions each at three levels on the yield of N.P.K were investigated. Observed response data (in triplicates) from experimental runs (Table 4) were used to develop models (Table 5) using least square techniques as described by Filli et al., (2010). The three (3) response variables (nitrogen N, phosphorus P and potassium P) were correlated with the independent variables using the second order polynomial as represented by Eq. (2). X_1, X_2, and X_3 represents the composting time, dosage ratio and moisture content respectively. The coefficient with one factor (X_1, X_2, and X_3) represents the sole effects of that particular factor, while coefficients with two factors (X_1X_2, X_1X_3 and X_2X_3) and those with second order terms (X_{11}, X_{22}, and X_{33}) represents the interaction between the three factors and the squared effect respectively. A positive value of the regression terms indicates a synergistic effect, while negative sign indicates an antagonistic effect Filli et al., (2010).

3.2.1. ANOVA analysis and model fitting

The Analysis of Variance (ANOVA) was utilized to translate the central composite design. The nitty gritty table of insights looks at the Sequential P-value, the Lack of fit P-value, the adjusted R-squared and the Predicted R-squared value. The synopsis of P-value shows that a quadratic model fitted the ANOVA examination and subsequently it was recommended (Table 5). The linear and 2FI models were not proposed. The Cubic model is constantly associated on the grounds that the CCD does not contain enough runs to help a full cubic model (Filli et al., 2010; Trinh and Kang, 2010). An significant level of 95% was utilized henceforth all terms whose P-value are <0.05 are viewed as significant terms.

The F-value tests were performed utilizing the ANOVA to ascertain the significance of each sort of model. Besides evaluating the significance, the adequacy of the models was evaluated by applying the lack-of-fit test. This test is utilized in the numerator in an F-trial of the null hypothesis and shows that a proposed model fits well or not. The test for lack of-fit contrasts the variation around the model with pure variation within the replicated observations. This test estimated the ampleness of the diverse models dependent on response surface investigation (Manpreet et al., 2011). Henceforth, the quadratic model with the most reduced insignificant model lack of fit was proposed.

Table 6 for N.P.K demonstrates the regression coefficients of the intercept, linear, quadratic and interactive terms of the models. The outcomes demonstrated that over 98% of the general framework factors can be explained by the quadratic model equations (Table 4). The significance of every coefficient in the models was checked from the P-value ($P < 0.05$) of the terms. The lower the models P-value (higher F-value) the better the significance of the input variable effect on the responses (Shrivastsvs et al., 2008).

From Table 6, the P-value for the models were (<0.0001) for nitrogen and additionally phosphorus and potassium which meant high significance in the prediction of the response factors and also the model appropriateness. The F-value was 59.37, 536.89 and 49.31 for nitrogen, phosphorus and potassium individually. These qualities were moderately high, accordingly showing that the models were exceptionally significant at above 95% confidence level. Their P-value built up the importance of the considerable number of coefficients as appeared in Table 6. From Table 6, every single liner term of time (X_1), dose ratio (X_2) and moisture content (X_3), the quadratic term of moisture content (X_3^2) and in addition the interactive term among time and dosage ratio (X_1X_2), time and moisture content (X_1X_3) and between dose ratio and moisture content were all significant with P-value < 0.05 for nitrogen. From Table 6 additionally, all linear terms of time (X_1), dose ratio (X_2) and moisture content (X_3), the quadratic term of time (X_1^2), dosage ratio (X_2^2), moisture content (X_3^2) and the interactive term among time and dose proportion (X_1X_2), time and moisture content (X_1X_3) and between dose ratio and moisture content were all significant with P-values < 0.05 for potassium, while every single liner term of time (X_1), dose ratio (X_2) and moisture content (X_3), the quadratic term of dose proportion (X_2^2) and also the interactive term among time and dosage ratio (X_1X_2), time and moisture content (X_1X_3) and between dosage ratio and moisture content (X_2X_3) were all significant with P-value under 0.05 for potassium. The values of the coefficient of determination (R^2) were 98.16% for nitrogen, 99.79% for phosphorus and 97.80% for potassium, accordingly a sign that the models fit the experimental data. Likewise, the values of the adjusted coefficient of regression (Adj R^2) and predicted coefficient of regression (Pred R^2) were 96.51% and 85.24% for nitrogen, 99.61% and 98.67% for phosphorus and 95.81% and 83.26% for potassium individually which indicates the model's significance and sensible accuracy of the fitted models respectively.

Table 5
Second order polynomial equation obtained for the three responses.

Response variables	Second order polynomial models	Regression coefficient	
		R^2	R^2_{adj}
N	$+9.31-0.24 * X_1-0.10 * X_2+0.13*X_3-0.19*X_1*X_2+0.15*X_1*X_3-0.11*X_2*X_3-0-0.34*X_3^2$	98.16	96.51
P	$+8.64-0.084*X_1-0.32*X_2+0.097*X_3-0.40*X_1*X_2+0.13*X_1*X_3-0.38*X_2*X_3-0.27*X_1^2-0.095*X_2^2-0.55*X_3^2$	99.79	99.61
K	$+4.45-0.086*X_1+0.11*X_2+0.17*X_3-0.16*X_1*X_2-0.21*X_1*X_3-0.28*X_2*X_3+0.37*X_3^2$	97.80	95.81

Table 6
Analysis of variance (ANOVA) for full quadratic model for the response variables.

Source	Sum of squares	df	Mean square	F value	P-value Prob > F
Nitrogen					
Model	3.45	9	0.38	59.37	< 0.0001
X_1-Time	*0.79*	*1*	*0.79*	*123.08*	*< 0.0001*
X_2-Dosage ratio	*0.15*	*1*	*0.15*	*22.66*	*0.0008*
X_3-Moisture content 0.24	*1*	*0.24*	*36.57*	*0.0001*	
X_1X_2	*0.29*	*1*	*0.29*	*44.75*	*< 0.0001*
X_1X_3	*0.18*	*1*	*0.18*	*27.89*	*0.0004*
X_2X_3	*0.11*	*1*	*0.11*	*16.39*	*0.0023*
X_1^2	*0.022*	*1*	*0.022*	*3.47*	*0.0921*
X_2^2	*2.022E-003*	*1*	*2.022E-003*	*0.31*	*0.5880*
X_3^2	*1.67*	*1*	*1.67*	*258.05*	*< 0.0001*
Residual	0.065	10	6.454E-003		
Lack of Fit	*0.28*	*10*	*0.028*	*0.19*	*0.9920*
Pure Error	*0.000*	*5*	*0.000*		
Cor Total	3.51	19			
Std. Dev.	0.080	R-Squared		0.9816	
Mean	9.06	Adj R-Squared		0.9651	
C.V. %	0.89	Pred R-Squared		0.8524	
PRESS	0.52	Adeq Precision		25.996	
Phoshorus					
Model	9.22	9	1.02	536.89	< 0.0001
X_1-Time	*0.096*	*1*	*0.096*	*50.48*	*< 0.0001*
X_2-Dosage ratio	*1.40*	*1*	*1.40*	*733.69*	*< 0.0001*
X_3-Moisture content 0.13	*1*	*0.13*	*67.10*	*< 0.0001*	
X_1X_2	*1.28*	*1*	*1.28*	*670.91*	*< 0.0001*
X_1X_3	*0.13*	*1*	*0.13*	*68.17*	*< 0.0001*
X_2X_3	*1.14*	*1*	*1.14*	*597.55*	*< 0.0001*
X_1^2	*1.04*	*1*	*1.04*	*543.13*	*< 0.0001*
X_2^2	*0.13*	*1*	*0.13*	*68.04*	*< 0.0001*
X_3^2	*4.38*	*1*	*4.38*	*2293.21*	*< 0.0001*
Residual	0.019	10	1.908E-003		
Lack of Fit	*0.014*	*5*	*2.799E-003*	*2.75*	*0.1453*
Pure Error	*5.083E-003*	*5*	*1.017E-003*		
Cor Total	9.24	19			
Std. Dev.	0.044	R-Square		0.9979	
Mean	8.02	Adj R-Squared		0.9961	
C.V. %	0.54	Pred R-Squared		0.9867	
PRESS	0.12	Adeq Precision		71.078	
Potassium					
Model	3.89	9	0.43	49.31	< 0.0001
X_1-Time	*0.10*	*1*	*0.10*	*11.46*	*0.0069*
X_2-Dosage ratio	*0.15*	*1*	*0.15*	*17.54*	*0.0019*
X_3-Moisture content 0.41	*1*	*0.41*	*46.62*	*< 0.0001*	
X_1X_2	*0.22*	*1*	*0.22*	*24.87*	*0.0005*
X_1X_2	*0.34*	*1*	*0.34*	*39.33*	*< 0.0001*
X_2X_3	*0.64*	*1*	*0.64*	*72.90*	*< 0.0001*
X_1^2	*0.019*	*1*	*0.019*	*2.12*	*0.1764*
X_2^2	*1.97*	*1*	*1.97*	*225.23*	*< 0.0001*
X_3^2	*7.276E-003*	*1*	*7.276E-003*	*0.83*	*0.3835*
Residual	0.088	10	8.758E-003		
Lack of Fit	*0.019*	*5*	*3.787E-003*	*2.96*	*0.1295*
Pure Error	*0.000*	*5*	*0.000*		
Cor Total	3.97	19			
Std. Dev.	0.094	R-Squared	0.9780		
Mean	4.71	Adj R-Squared	0.9581		
C.V. %	1.99	Pred R-Squared	0.8326		
PRESS	0.67	Adeq Precision	25.534		

Consequently, from these outcomes, it could be recommend that both liner and quadratic terms were the primary deciding factors for the yield of N.P.K. Adjusted R^2 is a measure of the variation around the mean clarified by the model, balanced for the quantity of terms in the model (Taran and Aghaie, 2015). The Adjusted R^2 diminished as the quantity of terms in the model increments if those extra terms do not increase the value of the model (Taran and Aghaie, 2015).

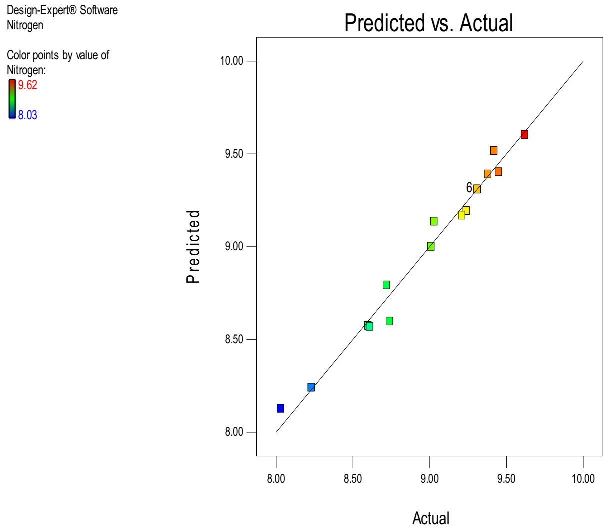
The tests for adequacy of the regression models, significance of individual of model coefficients and the lack of fit test were performed utilizing the same statistical package. The P-value were utilized as an apparatus to check the essentialness of every one of the coefficients, which thus are important to comprehend the example of the common collaborations between the test factors (Shrivastsvs et al., 2008). Higher the F-test value and small P-value indicates high significance of the relating coefficient (Taran and Aghaie, 2015). 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 (Taran and Aghaie, 2015). Hence, the adquate precision ratios of 25.996, 71.078 and 25.534 for N.P.K indicate adquate signal. This indicates that an adequate relationship of signal to noise ratio exists. The C.V called coefficient of variation which is defined as the ratio of the standard deviation of estimate to the mean value of the observed response is independent of the unit. It is also a measure of reproducibility and repeatability of the models (Chen et al., 2011). The calculations indicated the C.V value of 0.89% for nitrogen, 0.54% for phosphorus and 1.99% for potassium which showed that the model can be considered reasonably reproducible (because its CV was not >10%) (Chen et al., 2011). The response values obtained by inserting the independent values are the predicted values of the model. These values are compared to the actual experimental values. The result of this comparison is shown in the Table 7. From the table, it is seen that there is a close correlation between the actual experimental response and the predicted response. This confirms the effectiveness of the process for biofertilizer synthesis.

3.2.2. Model adequacy check

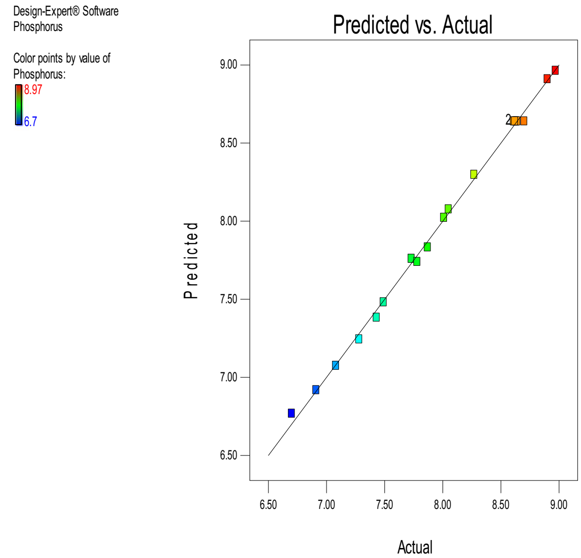
It is very important in RSM that the developed models (Table 5) provide an adequate approximation for application in real system, and there are principally two methods used for this check, these are graphical and numerical method Filli et al. (2010). The graphical technique considers the idea of the nature of the residuals (distinction between the observed values and its fitted) of the model while the numerical method utilizes the coefficient of determination (R^2) and adjusted R^2 (R^2_{adj}). For the most part, it is imperative to check the fitted model to guarantee that it gives the estimate to the genuine framework. On the off chance that the model does not demonstrate a sufficient fit, further examination and improvement of the fitted response surface may give

Table 7
Responses with predicted values of nitrogen for biofertilizer synthesis.

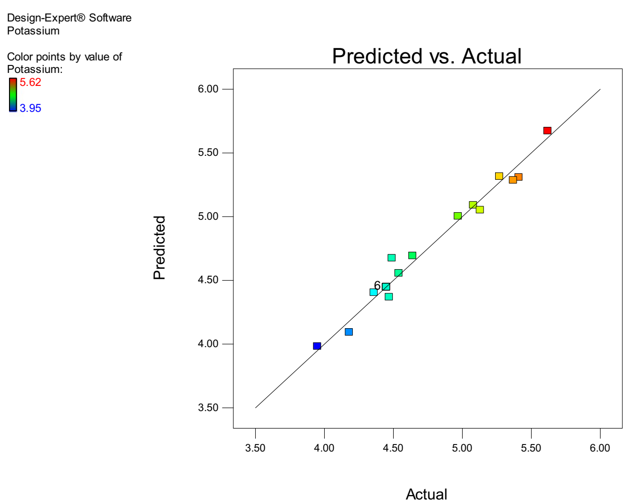
Standard order	Nitrogen		Phosphorus		Potassium	
	Actual value	Predicted value	Actual value	Predicted value	Actual value	Predicted value
1	9.01	9.00	7.43	7.38	3.95	3.98
2	8.74	8.60	7.73	7.76	4.54	4.56
3	9.45	9.40	8.27	8.30	5.62	5.67
4	8.23	8.24	8.05	7.07	4.97	5.00
5	9.24	9.19	7.08	8.08	5.41	5.31
6	9.38	9.39	8.97	8.96	5.13	5.05
7	9.03	9.14	7.49	7.48	5.37	5.29
8	8.62	8.57	6.70	6.77	4.47	4.37
9	9.62	9.60	8.01	8.02	4.64	4.69
10	8.72	8.79	7.78	7.74	4.36	4.40
11	9.21	9.52	8.90	8.91	5.27	5.32
12	8.03	8.06	7.87	7.83	5.08	5.09
13	8.61	8.13	6.91	6.92	4.18	4.09
14	9.31	8.57	7.28	7.24	4.49	4.60
15	9.28	9.31	8.70	8.64	4.45	4.45
15	9.33	9.31	8.61	8.64	4.44	4.45
17	9.31	9.31	8.63	8.64	4.43	4.45
18	9.27	9.31	8.63	8.64	4.46	4.45
19	9.32	9.31	8.64	8.64	4.45	4.45
20	9.31	9.31	8.62	8.64	4.43	4.45


Design-Expert® Software
Nitrogen
Color points by value of
Nitrogen:
9.62
8.03
Predicted vs. Actual
Predicted
Actual
10.00
9.50
9.00
8.50
8.00
8.00
8.50
9.00
9.50
10.00
6

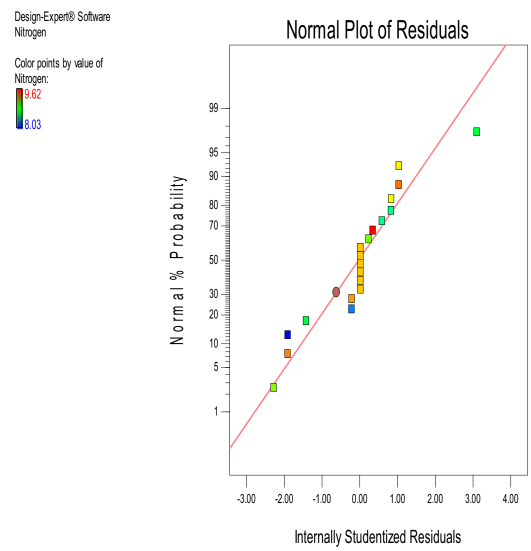
a


Design-Expert® Software
Phosphorus
Color points by value of
Phosphorus:
8.97
6.7
Predicted vs. Actual
Predicted
Actual
9.00
8.50
8.00
7.50
7.00
6.50
6.50
7.00
7.50
8.00
8.50
9.00
2

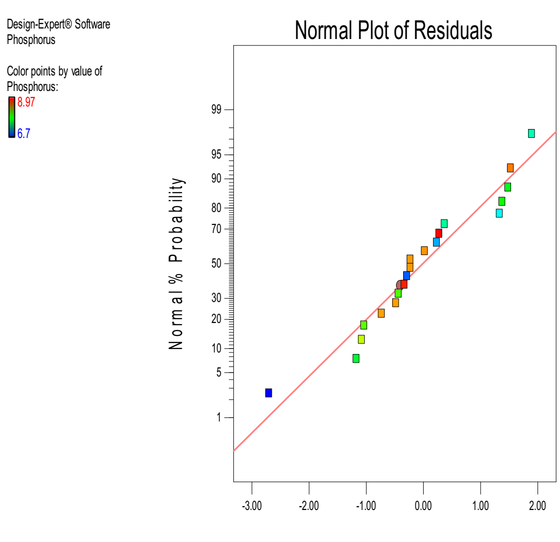
b


Design-Expert® Software
Potassium
Color points by value of
Potassium:
5.62
3.95
Predicted vs. Actual
Predicted
Actual
6.00
5.50
5.00
4.50
4.00
3.50
3.50
4.00
4.50
5.00
5.50
6.00
6

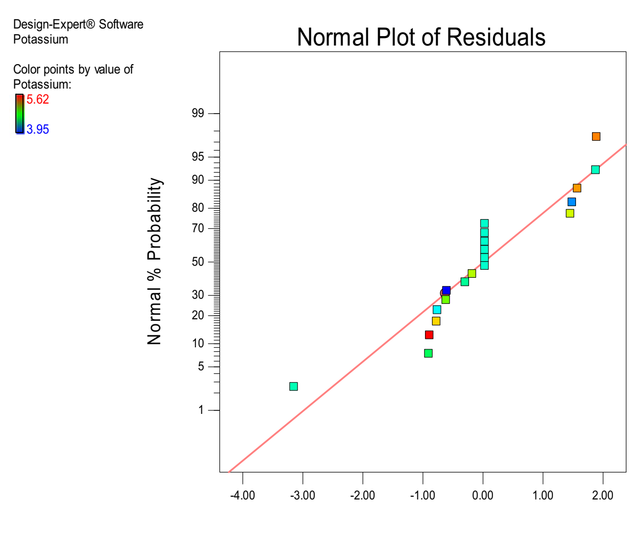
c


Design-Expert® Software
Nitrogen
Color points by value of
Nitrogen:
9.62
8.03
Normal Plot of Residuals
Normal % Probability
Internally Studentized Residuals
99
95
90
80
70
50
30
20
10
5
1
-3.00
-2.00
-1.00
0.00
1.00
2.00
3.00
4.00

d


Design-Expert® Software
Phosphorus
Color points by value of
Phosphorus:
8.97
6.7
Normal Plot of Residuals
Normal % Probability
Internally Studentized Residuals
99
95
90
80
70
50
30
20
10
5
1
-3.00
-2.00
-1.00
0.00
1.00
2.00

e


Design-Expert® Software
Potassium
Color points by value of
Potassium:
5.62
3.95
Normal Plot of Residuals
Normal % Probability
Internally Studentized Residuals
99
95
90
80
70
50
30
20
10
5
1
-4.00
-3.00
-2.00
-1.00
0.00
1.00
2.00

f

poor or deluding results as stated by Li and Fu (2005). The residuals from least square fits to assume a fundamental job in making a decision about model adequacy (Myers and Montgomery, 2002). Fig. 1A (a, b and c), shows the distribution of the predicted value against the actual experimental values for nitrogen, phosphorus and potassium. From the plots, each of the observed values was compared to the predicted values calculated from the models.

The regression coefficients of 98.16%, 99.79% and 97.80% observed between the predicted and real values for the response factors are proof that that the regression model can represent to the experimental data well. It could be seen that that the points on the diagrams were sensibly dispersed almost a straight line demonstrating that the fundamental supposition of typicality in this examination was proper and along these lines approve the models developed. The Normal plot of residuals as appeared in Fig. 1B (d, e and f), was utilized to check whether the points will pursue a straight line in which we presume that the residuals pursue a typical dissemination. It was seen that the points were firmly conveyed to the straight line of the plot. This affirms the great connection between the trial values and the predicted values of the response, however some little disperse like a "S" shape is constantly anticipated. This observation shows that the central composite design is well fitted into the model and thus can be used to perform the optimization operation for the process. Also, the straight line formed by the data points is an indication that neither response transformation is required nor there was any apparent problem with normality assumption of the regression model equations. This is in harmony with the report by Damirel and Kayan, (2012).

The R^2 measures how much of the observed variability in the experimental data could be accounted for by the models; while R^2_{adj} on the other hand modifies R^2 by taking into account the number of predictors in the model. R^2 and R^2_{adj} are calculated using Eqs. (4) and (5).

$$R^2 = \frac{Sum\ of\ square\ residual}{Model\ sum\ of\ square + Sum\ of\ square\ residual} \tag{4}$$

$$R^2_{adj} = 1 - \frac{n-1}{n-p}\left(1-R^2\right) \tag{5}$$

where n is the number of experimental runs, and p is the quantity of indicators in the model, not including the steady term. Kooche et al., (2009), recommended that for a decent fitted model, R^2 ought not be under 80%, while Chauhan and Gupta (2004), announced R^2 more noteworthy than 78% as worthy for fitting a model. In this examination, the models created showed R^2 going somewhere in the range of 97.80% and 99.79% while R^2adj extends somewhere in the range of 95.81 and 99.61% connoting fitness of the developed model equations in anticipating nutrient release in the compost during composting when the three independent factors are mathematically combined.

The R^2 and R^2_{adj} values are near unity. (Lee and Wang, 1997; Zaibunnisa et al., 2009), detailed that when R^2 is nearer to solidarity, the better the exact model fit the experimental data. It is not any more news that adding extra factor to the model will dependably build R^2, not considering of whether the extra factor is statistically significant or not. Consequently, a large R^2 does not always necessitate adequacy of the model. For this reason, Koocheki et al. (2009) declare that it is more fitting to utilize R^2_{adj} of over 90% to assess the model adequacy. Higher R^2_{adj} demonstrated that non-critical terms have not been incorporated into the model as obvious in this investigation. The general impression is that the residuals (Fig. 1) diffuse haphazardly in plain view, proposing that the fluctuation of the first perception is steady for all value of responses(Y). Since the plots in Fig. 1 are agreeable, it very well may be reasoned that the model is satisfactory to depict the mineralization of critical soil supplement amid composting of agricultural wastes for biofertilizer synthesis.

3.2.3. Factors and interactive effects on the mineralization of nitrogen during composting

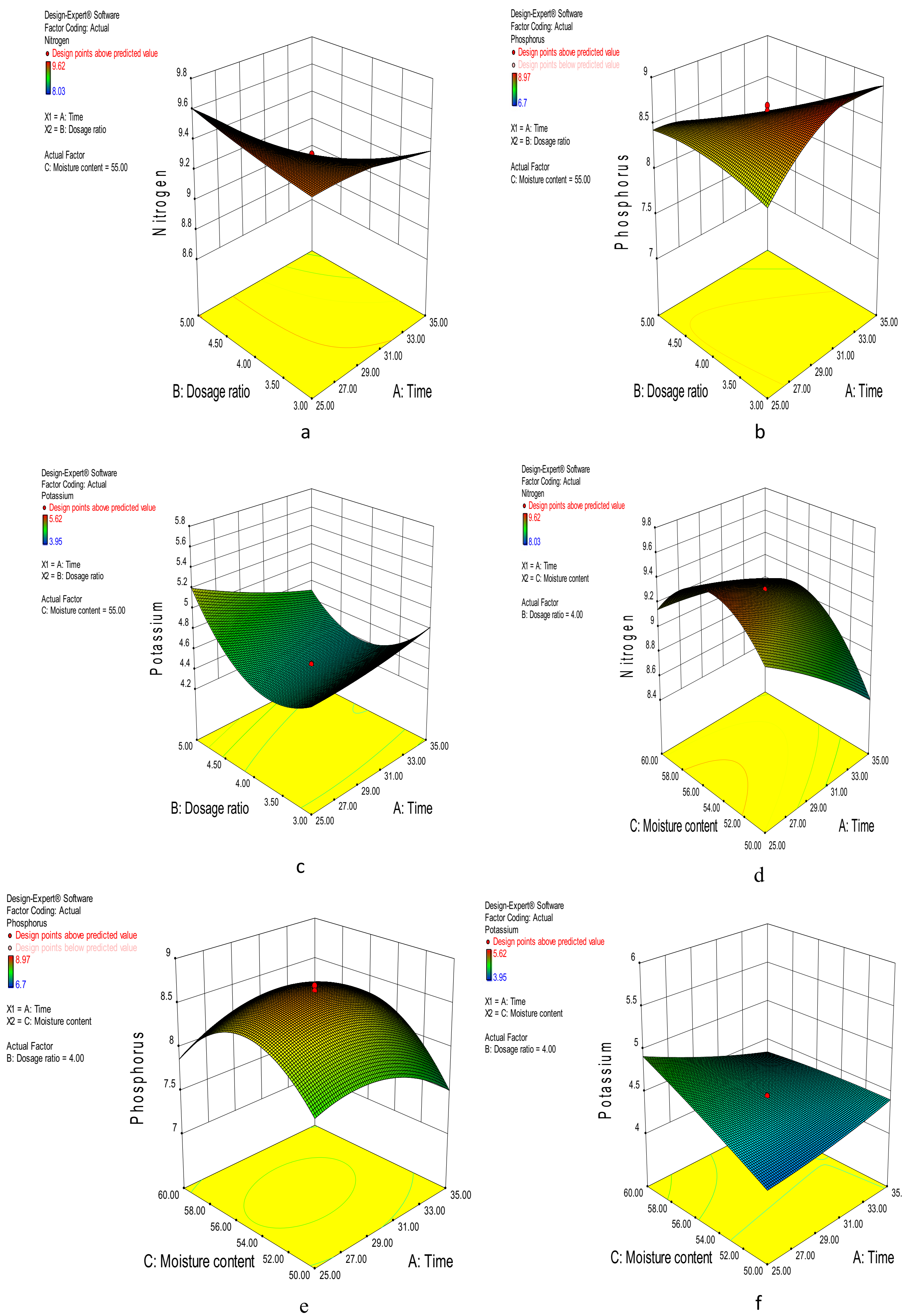
The interactive relationship between the independent and dependent variables are depicted by plotting 3-D graphs known as response surface graphs generated by the models. These plots were used to show the effect of process parameters on the yield of nitrogen, phosphorus and potassium (Lee and Wang, 1997). The linear, quadratic and interaction terms of the models (Table 5) were applied to create 3-D response surface graphs. Every margin on the graph denotes a specific value for the heights of the surface above the plane define for the combination of the levels of the independent variables (Liu et al., 2011). The 3-D response surfaces were generated by keeping one variables at its zero level (null point or midpoint) and carefully varying the other two variables within the experimental range.

Table 6 showed that the interaction of dosage ratio and time when the moisture content were kept constant at 55% were statistically significant as evident from the P-values ($P < 0.0001$ for nitrogen and phosphorus and $P < 00005$ for potassium). It was observed from Fig. 2A.(a-c), that as the dosage ratio were varied at different fractions, percentage nitrogen, phosphorus and potassium increases with increase in number of days (time) until it reaches an optimal point. Increase in both variable beyond the optimal point resulted to decrease in the percentage yield. At dosage ratio slightly above 5w:w and time above 33 days, the yield starts to decrease. The observation shows that dosage ratio and time has a significant effect on the mineralization of important soil nutrients from agro wastes during composting which is in harmony with the report by Gajalakshmi and Abbasi (2008). The shape of the contour lines in Fig. 2A.(a-c), is also an indication of strong interaction effect between dosage and time. The contours are somewhat curve which depicts the fact that the line will meet at a certain point and interaction would take place.

The interaction effect between moisture content and time were observed to be statistically significant at P-value ($P < 0.0004$ for nitrogen and $P < 0.0005$ for phosphorus) but not significant for potassium and was removed as shown in Table 6. It was observed from Fig. 2B. (d-f), that keeping the dosage ratio constant at dosage ratio of 4w: w, the yield of nitrogen and phosphorus increased by varying the moisture content between 50 and 60% with increase in number of days (time). The curve nature of the contour lines in Fig. 2B.(d-f), shows that interaction of the two factors is imminent and nutrient release certain. The percentage nitrogen and phosphorus was observed to have decreased at moisture content above 60% and moisture content below 50%. This trend is in harmony with the report by (Rasapoor et al., 2009; Stentiford, 1996) which states that excess moisture content impedes the entrance of oxygen to the compost system thereby changing the system from the desired aerobic co-fermentation to anaerobic co- fermentation; also low moisture content inhibits the growth and multiplication of microorganisms and hence prolongs the rate of nutrient release during composting.

Furthermore, the interaction effect of moisture content and dosage ratio were statistically significant at $P < 0.05$ (0.0023 for nitrogen, 0.0001 for phosphorus and potassium) as shown in Table 6. It was observed from Fig. 2C. (g), that the contour lines are somewhat parallel, which signifies poor interaction between moisture content and dosage ratio. From Fig. 2C.(h and i), the contour lines are mostly curve and not parallel to each other which is an indication of good interaction and positive influence on the yield. Keeping the time constant at 30 days, the yield of nitrogen, phosphorus and potassium increased at verifying moisture content and dosage ratio within the experimental range. The increase in the release of nitrogen at varying dosage ratio

Fig. 1. A: (a, b, c) Plots of predicted value vs experimental value for nitrogen, phosphorus and potassium. B: (d, e and f) Plots of normal probability plot of residual for nitrogen, phosphorus and potassium.



Design-Expert® Software
Factor Coding: Actual
Nitrogen
Design points above predicted value
9.62
8.03
X1 = A: Time
X2 = B: Dosage ratio
Actual Factor
C: Moisture content = 55.00
Nitrogen
B: Dosage ratio
A: Time
a
Design-Expert® Software
Factor Coding: Actual
Phosphorus
Design points above predicted value
Design points below predicted value
8.97
6.7
X1 = A: Time
X2 = B: Dosage ratio
Actual Factor
C: Moisture content = 55.00
Phosphorus
B: Dosage ratio
A: Time
b
Design-Expert® Software
Factor Coding: Actual
Potassium
Design points above predicted value
5.62
3.95
X1 = A: Time
X2 = B: Dosage ratio
Actual Factor
C: Moisture content = 55.00
Potassium
B: Dosage ratio
A: Time
c
Design-Expert® Software
Factor Coding: Actual
Nitrogen
Design points above predicted value
9.62
8.03
X1 = A: Time
X2 = C: Moisture content
Actual Factor
B: Dosage ratio = 4.00
Nitrogen
C: Moisture content
A: Time
d
Design-Expert® Software
Factor Coding: Actual
Phosphorus
Design points above predicted value
Design points below predicted value
8.97
6.7
X1 = A: Time
X2 = C: Moisture content
Actual Factor
B: Dosage ratio = 4.00
Phosphorus
C: Moisture content
A: Time
e
Design-Expert® Software
Factor Coding: Actual
Potassium
Design points above predicted value
5.62
3.95
X1 = A: Time
X2 = C: Moisture content
Actual Factor
B: Dosage ratio = 4.00
Potassium
C: Moisture content
A: Time
f

could be attributed to the component of the organic mixtures which has a great influence in the range of carbon to nitrogen ratio, which is the one of the key performance indicator in composting. Agro wastes must be blended at a certain dosage to maintain the carbon to nitrogen ratio from 15:1 to 35:1 for proper composting. López et al. (2010), suggested that C/N of around 20:1–35:1 are normally advisable, but good results have been obtained with values out of this range. In general, at higher C/N ratios (above 35:1), the composting process is thought to be delayed through lack of nitrogen, whereas smaller C/N ratios (<15:10) leads to excessive nitrogen loss and eventually to microbial toxicity due to high level of ammonia. Mixing of different materials is commonly necessary to achieve adequate C/N ratio for composting through proper dosage ratio (López et al., 2010).

3.2.4. Process optimization and validation of the composting process

As quoted by Josh et al. (2014), it is not possible to define a single optimum for a process since it can change depending on the level of other factors; however one of the optimum solutions was selected at desirability of 1 for the process composting. The consequences of the confirmation of the ideal numerical arrangement demonstrated that the ideal arrangement anticipated the genuine arrangement intently. The deviation of the test yield of nitrogen, phosphorus and potassium from the anticipated yield was a nearby match. The diagram of the predicted values against experimental (real values) as appeared in Fig. 1 likewise affirmed the closeness of the predicted and real value. The data points of the optimization runs falls along the inclining of the squared chart demonstrating the cozy relationship of the predicted and real points and all things considered, the quadratic model was satisfactory for the examination.

The developed model for the process parameters was streamlined utilizing response optimizer software that is available in design expert version 8.7.0.1. The software provides ideal answers for the input variable combinations. The optimization is additionally interactive and considers bargain among the different independent factors and the response(s) (Agu et al., 2015). The RSM was utilized to portray the connection between the process parameters and response factors (% yield) for the composting procedure. These process parameters are composting time, dosage ratio and moisture content while N.P.K is the response. The ideal setting is determined by characterizing the constraints and the objective function of the dependent factors. This way, the best values for both the input and response parameters are determined (Myers and Montgomery, 2002; Agu et al., 2015). In this research (work), the objective function of the response factors expands the rate yield of N.P.K. This is subject to the accompanying constraints: Composting time (time) (21.59 ≥ 38.41 days), dosage ratio (2.32 ≥ 5.68 w:w) and moisture content (46.59 ≥ 63.41%). The developed models were utilized by the response optimizer to provide the ideal outcomes for the responses and the independents factors.

The ideal conditions for the for maximum yield of nitrogen, phosphorus and potassium from the compost (mixtures of sawdust + sewage sludge + vegetable waste) concerning the proposed second order polynomial equations were: Composting time of 22 days, dosage ratio of 4 w:w and moisture content of 55% for nitrogen. At this condition, the predicted yield of nitrogen was 9.60%. Utilizing the ideal states of 22 days, 4w:w and 55%, the rate yield of nitrogen was tentatively validated. The outcome from the validation studies demonstrated a rate yield of 9.58%, which is relatively near the model's anticipated value. For phosphorus, the ideal condition was: Composting time of 35 days, dosage ratio of 3 w:w and moisture content of 60%. The anticipated rate yield of phosphorus was 8.96%. Utilizing the ideal state of 35 days, 3 w:w and 60%, the rate yield of phosphorus was validated. The outcomes from the validation experiment showed a rate yield of phosphorus was 8.91%, which moderately concurs with the model anticipated value. The ideal condition for potassium was: Composting time of 25 days, dosage ratio of 5w:w and moisture content of 50%. The anticipated rate yield for potassium at this condition was 5.67%. Utilizing the ideal state of 25 days, 5 w:w and 50%, the rate yield of potassium was tentatively validated. The consequence of the validation demonstrates that the rate yield was 5.69%, which was moderately near the model's anticipated value. The closeness of the validated value and predicted or anticipated rate yield of N.P.K shows the authenticity of the models. Additionally, a comparatively high R^2 value (98.16% for N, 99.79% for P and 97.80% for K) demonstrated the closeness between the tentatively validated experimental values and the anticipated or predicted values as appeared in Table 7. This further checks the accuracy of the proposed model.

3.3. Comparison of the results with prior results

The yield of nitrogen, phosphorus and potassium from the aforementioned examination was observed to be 9.62%, 8.97% and 5.62% respectively. The result of the percentage nitrogen was higher when compared with 4.7% nitrogen reported for the composting of mixture of sawdust and dewatered sewage sludge by Bazrafshan et al. (2006). The results was analyzed using Box Behnken Design and Validated at predicted value of percentage nitrogen 4.75% with regression coefficient of 95.6%. Also, 4.9%, 5.6% and 2.3% for nitrogen, phosphorus and potassium reported for the composting of mixture of sawdust plus chicken litter by Egbuna et al. (2016), which was the optimized results using CCD method of analysis and validated at predicted value of 4.95, 5.8, and 2.4 with regression coefficients of 92.5%, 94.2% and 91.7%. Moreover, 5.5%, 6.1% and 3.4% for nitrogen, phosphorus and potassium reported by Dumitrescu et al., (2009), after composting a mixture of sawdust plus sewage sludge plus dry leaves and analyzed with CCD and validated at the predicted values of 5.6%, 6.3% and 3.9% with regression cofficients of 91.8%, 93.6% and 90.8%. Nevertheless, after analysis with BBD, 7.84% nitrogen was reported by Olayinka and Adebayo (1989) after composting a mixture of sawdust plus cowdung and validated at the predicted value of 7.95% with regression coefficient of 96.7%, thus indicating the potential of the biofertilizer produced in this contest for commercial application. The difference in N.P.K yield obtained by prior researchers and that obtained in this research work could be attributed to factors such as substrate type (nature of the nitrogen rich agro-wastes), carbon to nitrogen ratio of the composting mixture, compsting time (process duration), degradability of the substrates and the nature of autochtonous microbes that aids the decomposition (Haug, 2009). Other perceived factors could be aeration rate (depending on rate of compost agitation), compost temperature and compost pH (Trinh and Kang, 2010).

4. Conclusion

The use of response surface methodology and central composite design was helpful in determination of the ideal working conditions for composting of organic wastes for biofertilizer synthesis. It was built up that the second order polynomial model was adequate to define and anticipate the process responses to variation of input variables within the experimental range. The validity of the models was demonstrated by fitting the estimations of the factors to the model equations and carrying out experiments utilizing the same values. The graphical optimization utilized to locate the ideal conditions for the composting of agro wastes was characterized by the composting time of 22 days, dosage

Fig. 2. A: (a, b and c), 3-D response surface plots showing the relationship between independent variables (time and dosage ratio) and dependent variables (nitrogen, phosphorus and potassium). B: (d, e and f) 3-D response surface plots showing the relationship between independent variables (moisture content and time) and dependent variables (nitrogen, phosphorus and potassium). C: (h, i and j) 3-D response surface plots showing the relationship between independent variable (moisture content and dosage ratio) and dependent variables (nitrogen, phosphorus and potassium).

ratio of 4w:w and moisture content of 55% with 9.62% yield of nitrogen, composting time of 35 days, dosage ratio of 3 w:w and moisture content of 60% with 8.97% yield of phosphorus and composting time of 30 days, dosage ratio of 6 w:w and moisture content of 55% with 5.62% yield of potassium. Characterization of the composite demonstrated its possibilities for commercial application on agricultural land.

Authors' contributions

Author	Contribution
Christian O. Asadu	Provided study materials, reagents, writing the initial draft, coordinated research activities and analyses of the data, review and editing
Samuel O. Egbuna	Writing the initial draft, provided funds and supervised the data analysis, reviewing and editing the manuscript
Thompson O. Chime	Provided funding, Coordinated research activities and supervised data analysis, reviewing and editing the manuscript
Chibuzor N. Eze	Provided funding, writing the initial draft and data analysis, reviewing and editing
Gordian O. Mbah	Provided funding and supervised the draft and data analysis
Dibia Kevin	Provided study materials, reagent, equipments, review and editing
Anthony C. Ezema	Provided reagents, study materials, data analysis

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