Optimizing coagulation–floculation processes with aluminium coagulation using response surface methods

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Abstract
A data-based multivariate method such as response surface methods and desirability function is considered advantageous for analysing coagulation treatment optimization. Thus, this study investigated the optimization of coagulation–floculation using response surface methodology. The parameters investigated were pH, alum dose and alkalinity. The optimum coagulation conditions for the individual responses: turbidity, colour, residual aluminium and phenanthrene were pH 7.0, alum dose 80.0 mg/L and alkalinity 80.0 mg/L; pH, 6.5, alum dose 70.0 mg/L and alkalinity 90.0 mg/L; pH, 7.0, alum dose 63.2 mg/L and alkalinity 80.0 mg/L and pH 6.2, alum dose 80.0 mg/L and alkalinity of 80.0 mg/L, respectively. The model equation derived from the optimization study was adequate for predicting the response values. The quadratic model was significant ($p < 0.0001$), and it had a high correlation ($R^2, 0.746–0.975$) and an insignificant (LOF, $p > 0.05$) lack of fit.

Keywords Coagulation · Flocculation · Central composite design · Optimization · Response surface methodology

Introduction
Raw water and process parameters are invariably complex, and their mutual interrelation is nonlinear. Variations in chemical composition of raw water and the physical features of coagulation–floculation phenomenon affect the water treatment processes. Several organic and inorganic compounds in a suspended, colloidal and dissolved form influence the coagulation–floculation process. Variable factors such as silicate, pH, temperature, natural process conditions, a dose of the coagulant, and the hydraulic dynamics of the treatment water flow play critical roles in water treatment. Optimization of coagulation processes depends on several factors, the most relevant being initial turbidity, pH, reagents (coagulant, adjuvant) dosage and type, system hydrodynamics in coagulation and floculation stages, temperature and alkalinity (Xiao et al. 2009; Shi et al. 2007; Miron et al. 2010). Over the period, the frequency and pattern of natural coagulation condition deterioration has resulted in the pollution of surface water, from anthropogenic, natural and climatic conditions (Bobadilla et al. 2019; Su et al. 2017; Khatri and Tyagi 2015; Edotepayi et al. 2017).

Jar test has been used as a technique to assess the suitability and efficiency of the choice and application of coagulant for water treatment (Water 2017; Saritha et al. 2017). However, this experimental process is limited; in determining the optimum conditions for treatment because the process does not explore the whole practical space for the interactions of all variable conditions for coagulation and it is time consuming and laborious to be carried out (Zainal-Abideen et al. 2012). In the conduct of jar test experiment, aluminium sulphate is the primary coagulant choice for water treatment because it is cheap, available, safe to handle and easy to use (Taylor et al. 2010; Klimiuk et al. 1999; Bérubé and Dorea 2008). Aluminium sulphate (alum) is naturally the critical parameter during coagulation treatment; however, the efficiency of coagulation solutions is limited by pH (6.2–7.2) (Daryabeigi Zand and Hoveidi 2015). After conducting the jar test experiment to assess the suitability and efficiency of a coagulant to be used in water treatment, one of the most
efficient statistical tools to be employed is the multivariate statistical technique.

The multivariate statistical technique for optimizing water coagulation treatment is advantageous because it determines the appropriate conditions, uses optimum conditions for coagulation and allows interactions among individual condition variables. Optimizing coagulation treatment requires suitable conditions using a multivariate statistical experimental method such as the response surface methodology (RSM) (Pinzi et al. 2010). RSM is extensively used to optimize pharmaceutical, bio-diesel, biochemistry and Agricultural production (Ghafari et al. 2009; Methodology 2011; Behera et al. 2018; Silva et al. 2011). Several researchers have used different types and experimental conditions and models for optimization (Zainal-Abideen et al. 2012; Bobadilla et al. 2019; Mäkelä 2017; Tolson et al. 2015). A compromise between optimization conditions of critical variables is essential for cost-effectiveness and water treatment reliability (Behera et al. 2016). Treated water particulate matter and organic compound removal are a function of crucial selected coagulation conditions such as pH, alkalinity and coagulant dose. Studies indicate that optimization of individual responses after treatment had different coagulation conditions for their efficient removal (Na et al. 2016).

Based on the importance of suitable pH conditions, alum dose and alkalinity on coagulation–flocculation condition, this study sought to optimize coagulation–flocculation process using alum to apply RSM. This study aims to determine the optimum pH, alum dose and alkalinity required to achieve an optimum coagulation–flocculation process using RSM. To realize the aim of the study, the study was premised on the following objectives: quantitatively determine the effects of the test variables on the response outcome; assess interrelationship among test variables; evaluate the combined effect of all the test variables on the response outcome and to validate the model adequacy in predicting coagulation efficiency.

Materials and methods

To determine the optimum coagulation conditions and optimize the coagulation–flocculation process, aluminium sulphate solution at varying dosages between 60 and 110 mg/L from a daily stock solution of 1% strength was simulated. The process used a variable-speed jar tester from Phipps and Bird equipped with six flat impellers and illuminated base. Aluminium sulphate hydrated (Al₂SO₄·18H₂O), coagulant of choice supplied from KEMIRA.BV with specification for aluminate 17.2% and density, 1.0291 g/cm. All the reagents used were of analytical grade, and the solution pH and alkalinity were adjusted with 0.1 M NaOH or HCl and NaHCO₃. Coagulation test was performed in a 1 L cylindrical beaker at 25 ± 1 °C. Raw water was collected at the inlet to the 1150 mm ductile iron pipe for the jar test. Grab sampling type in a sequential process was used for both the raw water and settled water after coagulation–flocculation and clarification. The source of water used for the experiment was collected from the Weija Reservoir in Accra, Ghana. The Weija reservoir is characterized by high colour, turbidity and conductivity levels. Throughout the work, rapid mixing was conducted in 250RPM/30 s in order to provide a uniform dispersion of the coagulant throughout the influent water, and slow mixing at 30RPM for 15 min so as to bring the contacts between the finely divided destabilized matters formed during the rapid mixing (Fig. 1). In the jar test simulation process, settling period of 30 min was adopted to conform to the standard jar test simulation mechanism. After settling, the supernatant samples were collected using a pipette about 2 cm below the water surface to measure turbidity, colour, residual aluminium and poly aromatic hydrocarbons (PAHs, ASTM-D2035 2013; Water 2003; Estate n.d.).

The jar test simulation technique was conducted according to the guidelines of the Standard Methods for the Examination of Water and Wastewater (APHA 2017, 23rd Edition) and the USEPA Method 8270D. During the jar test analysis, the water quality parameter analysed included pH (should be optimized to maximize the removal of impurities present in raw water), alkalinity (high alkalinity is preferred for coagulation since it tends to have more positively charged ions to interact with the negatively charged colloids), aluminium (the dosage of alum, which represents least turbidity, gives optimum coagulant dosage, O.C.D.), colour, temperature, and poly aromatic hydrocarbon (PAH)-particularly phenanthrene. Samples for PAHs analysis were collected in a 1 L amber glass bottle with a Teflon screw cap recommended for PAHs sample before analysis and transported at 4 °C to the analytical laboratory for PAHs extraction. The solid phase extraction (SPE) was used for isolating targeted organic analyte from the water samples (EPA Method 8310). Before extraction, the SPE column was conditioned with 5 mL dichloromethane (DCM) and 5 mL methanol contiguously three times repetition under vacuum followed with 5 mL of deionised water. Before sample extraction, 10 mL of deionised water was spiked with the standard solution PAHs, (0.005 mg/L and subsequently passed through the column with the slow flow rate of 1 mL/min. The sample was extracted, and cartridge dried under full vacuum. Analytes were eluted with 15 mLs ethyl acetate thrice and freeze-dried using nitrogen gas. Elute was further constituted to 4 mL volume by adding 2 mLs of ethyl acetate and further dried with magnesium sulphate. After allowing 45 min contact time through shaken with the drying agent, the sample was centrifuged at 400RPM/4 min, and 1 mL was decanted and filtered into a vial through a 0.45-micron filter pore and submit to the laboratory to quantify the amount of analyte in the sample using GC/MS (SCHIMUDZI 4200) in line with USEPA 16, 8270 PAH method of analysis.
linearity range for the determination of 16 compounds of PAHs by GC/MS was 0.05 μg/L with correlation factor from 0.9959 to 0.9999. The one-factor variable approach was used (Zainal-Abideen et al. 2012; Benson n.d.), whilst the detection limit for SPE was in the range of 0.05–0.55 mg/with good recovery of 65.21–99.32%.

**Experiment design approach**

In the experimental approach using the one-factor variable, one coagulation conditions factor is varied with other factors being made constant (Zainal-Abideen et al. 2012; Benson n.d.). The coagulation conditions variables used in this experiment are alum dose, pH and alkalinity. The jar test experiment runs the factors in the sets of six jars and repetition for others. The pH and alkalinity of the solution were fixed at natural water characteristics to determine the optimum dose of the coagulant using different dosages of 60 mg/L, 70 mg/L, 80 mg/L, 90 mg/L, 100 mg/L and 110 mg/L. The second set of experiment was conducted using the best alum dosage of the first jar test run as constant and alkalinity values falling between 70 and 120 at ten unit’s intervals for the jar test. From the experiment, levels and ranges of the respective independent variables for pH (6.5–7.5) at 0.5-unit interval, alum dose (70–90 mg/L) at 10.0-unit interval and alkalinity (70–90 mg/L) at 10 unit interval were defined using \( \alpha = (NF)^{1/4} \), where N.F. is the number of points in the cube portion of the design \( N_F = 2^k \), \( k \) is the number of factors. Therefore, \( \alpha \) is equal to \( (2^3)^{1/4} = 1.682 \), according to Eq. 2. To obtain the axial points values are given by the equation.

The laboratory conditions under which the experiment was conducted were at standard temperature and pressure (STP). A pH meter (HACH-HQ 40d), turbidimeter (HACH-2100), spectrophotometer (HACH- DR-6000), AES-MS Spectrophotometry-(MP 4200, Agilent) and GC/MS -PBM (Schimudzi) were test instrument used in measuring parameters under study. The parameters are measured electronically except alkalinity which was done by titration using sulfuric acid. All tests were done in situ and observing strict compliance to preservation conditions with a reasonable sample custody requirement. The methods and processes went through strict Quality Control/Quality Assurance Compliance, as prescribed by the Standards Methods, 2017. The response surface methodology uses the second-factorial central composite design (CCD) in the
experimental design (Book 2016; Edokpayi et al. 2017; Zainal-Abideen et al. 2012; Behera et al. 2018; Ghafari et al. 2009). A desirability function is used to optimize multiple response processes under selected optimum conditions (Mang et al. 2015; Pinzi et al. 2010; Bobadilla et al. 2019). The number of experimental runs obtained at each number of factors is given in Eq. 1.

\[ N + 2^n + 2n + nc \]  

where \( N \) is the number of runs, \( n \), the number of factors and \( nc \), is the number of centre points. A total of 20 experiments in accordance with a \( 2^3 \) full factorial CCD, comprising eight factorial points (coded to the usual \( \pm 1 \) notation), with six axial points (\( \pm \alpha, 0, 0 \), \( 0, 0, \pm \alpha \)), and six replicates at the centre points (0, 0, 0) were conducted.

The general quadratic equation used in the RSM in analysing the coagulation treatment optimization is shown in Eq. 2:

\[
\hat{y} = \beta_0 + \beta_1X_1 + \beta_2X_2 + \beta_3X_3 + \beta_{12}X_1X_2 + \beta_{13}X_1X_3 + \beta_{23}X_2X_3 + \beta_{11}X_1^2 + \beta_{22}X_2^2 + \beta_{33}X_3^2
\]

**Results and discussion**

One factor at a time jar analysis and response outcomes on coagulation conditions for the removal efficiency of turbidity and colour is indicated in Table 1. The lowest turbidity removal was 65.7% at a pH of 6.0 and dosage of 80 mg/L alum at 90 mg/L alkalinity. The pH setting of 7.5 resulted in 82.3% turbidity removal. Color removal of 89.1% was maximum at a dose of 80 mg/L and a pH of 6.5. In contrast, 77.3% removal of turbidity at higher pH of 8.5 and the same dosage of 80 mg/L alum at 90 mg/L alkalinity was observed. An indication that the conditions for turbidity and colour removal are different and one best state for one contaminant does not favour removing the other (Saritha et al. 2017; Duan and Gregory 2003). A range of coagulation conditions will be applicable to favour particulate matter removal in the pH range of 6.5 and 7.5, alkalinity of 80–90 mg/L and dosage between 80 and 90 mg/L of alum. The coagulation removal mechanism for the contaminants is different. Turbidity removal occurred best at moderate pHs between 7.0 and 8.5 which also supports sweep flocculation mechanism. In contrast, charge neutralization at lower pHs between 6.0 and 6.9 supports colour removals. The conventional experimental jar test cannot give one desired variable for different contaminants. Hence, it does not explore all the testing space to bring the best removal of pollutants (Zainal-Abideen et al. 2012; Farhaoui and Derraz 2016).

The three (3) selected factors (pH, alum dose and alkalinity) of the coagulation condition with their ranges and levels obtained from the OFAT analysis are shown in Table 2. The best conditions determined from the one variable factor analysis give the coagulant dosage, 80 mg/L, alkalinity 80 mg/L and pH 7 used as central values. A range difference of 0.5

| RUN | Raw water Turbidity (NTU) | R. water colour (Pt. Co) | pH | Alum dosage (mg/L) | Alkalinity (mg/L) | Clarified Turbidity (NTU) | % Turbidity removal | Colour (Pt.Co) | % Colour removal |
|-----|--------------------------|--------------------------|----|-------------------|------------------|------------------------|-------------------|---------------|-----------------|
| OFAT 1 | 14.1 | 45.0 | 7.5 | 60.0 | 90.0 | 3.8 | 73.0 | 6.8 | 84.9 |
| OFAT 2 | 14.1 | 45.0 | 7.5 | 70.0 | 90.0 | 3.3 | 76.6 | 6.5 | 85.6 |
| OFAT 3 | 14.1 | 45.0 | 7.5 | 80.0 | 90.0 | 2.5 | 82.3 | 6.4 | 85.8 |
| OFAT 4 | 14.1 | 45.0 | 7.5 | 90.0 | 90.0 | 2.3 | 83.7 | 5.9 | 86.9 |
| OFAT 5 | 14.1 | 45.0 | 7.5 | 100.0 | 90.0 | 2.8 | 80.1 | 6.1 | 86.4 |
| OFAT 6 | 14.1 | 45.0 | 7.5 | 110.0 | 90.0 | 2.9 | 79.3 | 5.4 | 88.0 |
| OFAT 7 | 14.1 | 45.0 | 7.5 | 120.0 | 90.0 | 4.8 | 65.7 | 5.4 | 88.0 |
| OFAT 8 | 14.1 | 45.0 | 7.5 | 130.0 | 90.0 | 3.6 | 74.2 | 4.9 | 89.1 |
| OFAT 9 | 14.1 | 45.0 | 7.5 | 140.0 | 90.0 | 2.9 | 79.4 | 5.1 | 88.7 |
| OFAT 10 | 14.1 | 45.0 | 7.5 | 150.0 | 90.0 | 3.3 | 76.6 | 6.3 | 86.0 |
| OFAT 11 | 14.1 | 45.0 | 7.5 | 160.0 | 90.0 | 3.1 | 77.9 | 6.9 | 84.7 |
| OFAT 12 | 14.1 | 45.0 | 7.5 | 170.0 | 90.0 | 3.7 | 73.8 | 10.2 | 77.3 |
| OFAT 13 | 14.1 | 45.0 | 7.5 | 180.0 | 90.0 | 4.2 | 70.5 | 6.2 | 86.2 |
| OFAT 14 | 14.1 | 45.0 | 7.5 | 190.0 | 90.0 | 3.3 | 76.7 | 6.4 | 85.8 |
| OFAT 15 | 14.1 | 45.0 | 7.5 | 200.0 | 90.0 | 2.6 | 81.8 | 6.1 | 86.4 |
| OFAT 16 | 14.1 | 45.0 | 7.5 | 210.0 | 90.0 | 2.9 | 79.3 | 5.6 | 87.6 |
| OFAT 17 | 14.1 | 45.0 | 7.5 | 220.0 | 90.0 | 3.1 | 77.8 | 5.8 | 87.1 |
| OFAT 18 | 14.1 | 45.0 | 7.5 | 230.0 | 90.0 | 3.7 | 74.1 | 6.2 | 86.2 |
for pH, 10 mg/L for alum dose and 10.0 mg/L for alkalinity were selected. The ranges and levels for pH, alum dose range and alkalinity are shown in Table 2—the tool assigned the values, codes based on the designed purposes for the factorial experiment.

The values representing the independent factors and their corresponding values showing the axial points are shown in Table 3. Each numerical factor is a set to 5 levels; plus and minus alpha (axial point), plus and minus (factorial points) and the centre (Behera et al. 2018; Book 2016).

The impurity residual removal results at the experimental points are indicated in Table 4. Most factorial runs yielded results which comply with the water quality regulatory standards for settled water except for run 5, 6, 15, 16 and 19. Based on the RSM table's direct reading (Table 4), run 4 was decided as the optimum coagulation condition as it supernatant water quality meets compliance requirements with better responses of interest (turbidity (1.074NTU), colour (1.975Pt.Co), residual aluminium (0.009 mg/L) and phenanthrene (2.213 µg/L). Although run 2, 8 and 11 produced even lower turbidity in their settled water than in run 4 and satisfies the compliance limits for colour, residual aluminium and phenanthrene regulatory conditions in a whole their responses come second to run 4. Additionally, run 4

| Table 2 | Experimental coagulation condition (independent variable factors) ranges and levels |
|---------|----------------------------------------------------------------------------------|
| CODES   | −1.0 0.0 1.0                                                                        |
| A pH units | 6.5 7.0 7.5                                                                          |
| B Alkalinity (mg/L) | 70.0 80.0 90.0                                                                      |
| C Coagulant dose (mg/L) | 70.0 80.0 90.0                                                                     |

| Table 3 | Experimental range and levels for rotatable central composite design |
|---------|---------------------------------------------------------------------|
| Factors               | $\alpha$ (−1.31607) | −1 | 0 | +1 | $\alpha$ (+1.31607) |
|                      | Axial point | Lower level | Central point | Upper level | Axial point |
| $X_1$ (pH)           | 6.342       | 6.50       | 7.00       | 7.50       | 7.658       |
| $X_2$ (alum dosage)   | 66.839      | 70.00      | 80.00      | 90.00      | 93.161      |
| $X_3$ (alkalinity)    | 66.839      | 70.00      | 80.00      | 90.00      | 93.161      |

| Table 4 | Full factorial experimental design and responses for the experimental variables |
|---------|----------------------------------------------------------------------------------|
| Std | Run | Factors | Responses |
|      |     | A:pH | B:Alum dose (mg/L) | C:alkalinity (mg/L) | Turbidity (NTU) | Colour (Pt.Co) | Residual Aluminium (mg/L) | Phenanthrene (µg/L) |
| 2   | 1   | 7.5  | 70            | 70            | 1.648         | 2.11          | 0.04                  | 3.832               |
| 14  | 2   | 7    | 80            | 96.82         | 0.911         | 1.961         | 0.014                 | 2.163               |
| 10  | 3   | 7.84 | 80            | 80            | 1.984         | 2.084         | 0.028                 | 3.418               |
| 11  | 4   | 7    | 63.18         | 80            | 1.074         | 1.975         | 0.009                 | 2.213               |
| 5   | 5   | 6.5  | 70            | 90            | 2.113         | 1.21          | 0.011                 | 2.143               |
| 3   | 6   | 6.5  | 90            | 70            | 2.251         | 1.364         | 0.012                 | 2.257               |
| 6   | 7   | 7.5  | 70            | 90            | 1.136         | 2.319         | 0.008                 | 3.812               |
| 12  | 8   | 7    | 96.82         | 80            | 0.921         | 1.842         | 0.012                 | 2.682               |
| 16  | 9   | 7    | 80            | 80            | 1.286         | 2.527         | 0.043                 | 2.254               |
| 8   | 10  | 7.5  | 90            | 90            | 1.11          | 2.14          | 0.07                  | 3.491               |
| 4   | 11  | 7.5  | 90            | 70            | 0.94          | 2.01          | 0.04                  | 3.62                |
| 20  | 12  | 7    | 80            | 80            | 1.286         | 2.527         | 0.043                 | 2.254               |
| 13  | 13  | 7    | 80            | 63.18         | 1.416         | 2.38          | 0.06                  | 3.587               |
| 17  | 14  | 7    | 80            | 80            | 1.286         | 2.257         | 0.043                 | 2.254               |
| 1   | 15  | 6.5  | 70            | 70            | 2.331         | 2.168         | 0.03                  | 2.161               |
| 9   | 16  | 6.16 | 80            | 80            | 2.872         | 1.262         | 0.009                 | 2.001               |
| 18  | 17  | 7    | 80            | 80            | 1.286         | 2.527         | 0.023                 | 2.254               |
| 15  | 18  | 7    | 80            | 80            | 1.286         | 2.527         | 0.043                 | 2.254               |
| 7   | 19  | 6.5  | 90            | 90            | 2.412         | 1.265         | 0.009                 | 2.119               |
| 19  | 20  | 7    | 80            | 80            | 1.286         | 2.527         | 0.043                 | 2.854               |
provides efficient consumption of chemicals (pH: 7.0, alum dosage: 63.18 mg/L and alkalinity 80.0 mg/L as CaCO₃) during coagulation–flocculation in the experimental design run.

A diagnostic adequacy checks to test the proposed model using ANOVA indicated in Table 5. The model derived is significant at 95% confidence level. The turbidity and colour removal model satisfies reproducibility as their CV values are less than 10%. The removal model for residual aluminium and phenanthrene falls short with CV above 10%. The slightly higher variability for residual aluminium and phenanthrene is coming from extra residual load from the coagulant and their negligible initial concentration (Oehlert 2003). Table 5 confirms the predictive model's significance ($p < 0.05$). It also explains the improved removal for the water quality response parameters after coagulation–flocculation on the application of RSM indicated by the $p$ values < 0.0001 turbidity, < 0.0003 colour, < 0.0381 residual aluminium and < 0.0028 phenanthrene, respectively.

Estimates of the multiple regression equation for responses as a function of the factors and their significance are presented in Table 5. The coefficient estimates represent the expected changes in the response per unit change in factor value when all remaining factors are held constant. The intercept in an orthogonal design is the overall average response of all the runs, and its significance denotes a good model. The coefficients are adjustable around the average based on the factor settings. In the table, the coefficient estimates for the linear response are significant for the variables pH (A), alum dose (B) and alkalinity (C). The square and the interactive terms of the variables had mixed significance and insignificance (Table 6).

The predicted interactive effects of alum and alkalinity in the removal efficiency of turbidity are indicated in Table 6. The results in Table 6 show that the three operation conditions play a significant ($p$ value < 0.05) role and has positive results in the removal of turbidity (Iriarte-Velasco et al. 2007; Iriarte-Velasco et al. 2007).

Equations derived from the quadratic model in Table 6 are in terms of the coded factors (A, B and C). The equation is used to make predictions about the response for given levels of each factor shown in Table 7. The higher levels of factors are coded as +1, and the low levels are coded as −1. The coded equation is useful for identifying the relative impact of factors by comparing the factor coefficients. The coded factor A, which represents pH, shows appearance in all the equations having effects on all the responses in the linear and quadratic models. They indicate that pH is significantly an influence factor and impacts the coagulation–flocculation process for contaminant removal (Trinh and Kang 2011).

| Response | Model F-value | Model $p$ value | Model Prob > F | R2 | Adjustable R2 | Adequate precision (AP) | SD | CV | Mean | PRESS |
|----------|--------------|----------------|---------------|----|---------------|-------------------------|----|----|------|--------|
| Turbidity | 43.95 | < 0.0001 | 0.128 | 0.9753 | 0.9532 | 24.2358 | 0.125 | 8.09 | 1.54 | 1.27 |
| Colour | 11.5 | 0.0003 | 0.0598 | 0.9119 | 0.8327 | 10.3389 | 0.184 | 9 | 2.05 | 2.77 |
| Residual Aluminium | 3.31 | 0.0381 | 0.0762 | 0.7485 | 0.5222 | 5.8786 | 0.013 | 43.9 | 0.0295 | 0.012 |
| Phenanthrene | 6.96 | 0.0028 | 0.1352 | 0.8623 | 0.7383 | 8.4457 | 0.341 | 12.7 | 2.68 | 7.05 |

LOF lack of fit, SD standard deviation, CV coefficient of variation, PRESS prediction error sum of squares

Table 5 ANOVA results for response parameters in RSM jar test

| Factor | Turbidity | Colour | Res.Aluminium | Phenanthrene |
|--------|-----------|--------|--------------|--------------|
| Intercept | 118.086 | < 0.0001 | − 57.855 | 0.000 | 0.379 | 0.038 | 48.448 | 0.003 |
| A-pH | −36.039 | < 0.0001 | 17.127 | 0.000 | 0.159 | 0.020 | − 9.198 | < 0.0001 |
| B-Alum Dosage | 0.303 | 0.121 | 0.189 | 0.083 | − 0.005 | 0.319 | 0.036 | 0.895 |
| C-Alkalinity | 0.029 | 0.031 | − 0.215 | 0.069 | − 0.019 | 0.083 | − 0.487 | 0.089 |
| AB | − 0.024 | 0.022 | 0.012 | 0.389 | 0.002 | 0.049 | − 0.015 | 0.545 |
| AC | − 0.007 | 0.438 | 0.035 | 0.023 | 0.001 | 0.597 | 0.000 | 0.994 |
| BC | 0.001 | 0.013 | 0.001 | 0.166 | 0.000 | 0.059 | 0.000 | 0.817 |
| A² | 2.683 | < 0.0001 | − 1.444 | 0.000 | − 0.024 | 0.242 | 0.841 | 0.135 |
| B² | − 0.002 | 0.008 | − 0.002 | 0.009 | 0.000 | 0.054 | 0.000 | 0.657 |
| C² | − 0.001 | 0.238 | − 0.001 | 0.315 | 0.000 | 0.373 | 0.003 | 0.040 |

Table 6 Estimate of the model equation for the response results
The residual aluminium response is a function of the amount of aluminium dose in the solution and depends on the conditions provided by pH and alkalinity (Yukselen and Gregory 2004). Here, more than one mechanism is employed to remove residual aluminium.

Equation (6) shows that phenanthrene removal is impacted significantly \((p < 0.05)\) by the linear effect of pH. However, the interactive effect of the factors was less significant (Table 5). Phenanthrene removal is related to pH effects (Trinh and Kang 2011; Hurst et al. 2004). Phenanthrene colloids by their nature make it possible to be coagulated due to their high molecular weight and hydrophobic character (Yukselen and Gregory 2004). They are not considered part of true water-soluble fraction and can be removed from the water by the application of charge neutralization (Yukselen and Gregory 2004). Solution pH that influences phenanthrene colloidal charges contributes significantly to the removal by charge neutralization.

\[
\text{Turbidity} = \frac{118.08 - 36.079 A + 0.029 C}{0.024 A + B - 0.001 B + C - 2.683 A^2 - 0.002 B^2} \quad (3)
\]

\[
\text{Color} = \frac{-57.855 + 17.127 A + 0.035 A + C - 1.444 A^2 - 0.002 B^2}{0.002} \quad (4)
\]

\[
\text{Aluminium. Res} = \frac{0.379 + 0.159 A + 0.002 A + B}{0.003} \quad (5)
\]

\[
\text{Phenanthrene} = \frac{48.448 - 9.198 A + 0.003 C^2}{0.003} \quad (6)
\]

\[
\begin{align*}
\text{Equation (4)} \quad &\text{is colour removal model; the effects of the factors were similar to the removal of turbidity with the linear (pH), interactive (pH-alkalinity) and quadratic effects (pH^2 and alum dose^2) to be significant. The significance of the interactive effect of pH and alkalinity plays an essential role in colour removal at a lower dose at values 7.0 pH units, 63.2 mg/L coagulant and 80 mg/L as CaCO}_3 \text{ provide significant impetus for sweep flocculation process. Turbidity removal enhanced by precipitated hydroxide molecule from alkaline also facilitates effective coagulation.}
\end{align*}
\]

\[
\begin{align*}
\text{Equation (5)} \quad &\text{presents a good model for residual aluminium response. As indicated by the equation, the linear model effects of pH and alkalinity for the coagulation solution are substantial, with p values of 0.013 and 0.038, respectively. The interactive effects of pH and alum dosage and alum dosage and alkalinity on residual aluminium removal are significant with p values of 0.030 and 0.032, respectively. The quadratic effects of alum dose were significant \((p < 0.05)\). The residual aluminium response is a function of the amount of aluminium dose in the solution and depends on the conditions provided by pH and alkalinity (Yukselen and Gregory 2004). Here, more than one mechanism is employed to remove residual aluminium.}
\end{align*}
\]

\[
\begin{align*}
\text{Equation (6)} \quad &\text{shows that phenanthrene removal is impacted significantly \((p < 0.05)\) by the linear effect of pH. However, the interactive effect of the factors was less significant (Table 5). Phenanthrene removal is related to pH effects (Trinh and Kang 2011; Hurst et al. 2004). Phenanthrene colloids by their nature make it possible to be coagulated due to their high molecular weight and hydrophobic character (Yukselen and Gregory 2004). They are not considered part of true water-soluble fraction and can be removed from the water by the application of charge neutralization (Yukselen and Gregory 2004). Solution pH that influences phenanthrene colloidal charges contributes significantly to the removal by charge neutralization.}
\end{align*}
\]

\[
\begin{align*}
\text{Turbidity} = \frac{118.08 - 36.079 A + 0.029 C}{0.024 A + B - 0.001 B + C - 2.683 A^2 - 0.002 B^2} \quad (3)
\end{align*}
\]

\[
\begin{align*}
\text{Color} = \frac{-57.855 + 17.127 A + 0.035 A + C - 1.444 A^2 - 0.002 B^2}{0.002} \quad (4)
\end{align*}
\]

\[
\begin{align*}
\text{Aluminium. Res} = \frac{0.379 + 0.159 A + 0.002 A + B}{0.003} \quad (5)
\end{align*}
\]

\[
\begin{align*}
\text{Phenanthrene} = \frac{48.448 - 9.198 A + 0.003 C^2}{0.003} \quad (6)
\end{align*}
\]

\[
\begin{align*}
\text{Table 7} \quad &\text{Contaminant removal results at individual optimum desirable conditions}
\end{align*}
\]

| Factors          | Low   | High  | Optimum |
|------------------|-------|-------|---------|
| pH               | 6.5   | 7.5   | 6.9     |
| Alum Dosage(mg/L)| 70    | 90    | 71      |
| Alkalinity (mg/L)| 70    | 90    | 80      |
| Turbidity (NTU)  | 0.911 | 2.872 | 1.2115  |
| Colour (Pt.Co)   | 1.21  | 2.527 | 2.1272  |
| Res.Aluminium(mg/L)| 0.008 | 0.07  | 0.013   |
| Phenanthrene (µg/L)| 2.001 | 3.832 | 2.245   |

The pH effect is consistent with the study conducted by Lanciné et al. (2008). That, pH determines at the same time the electric charge of both organic and inorganic colloids and it is essential in the hydrolysis of aluminium coagulant, as well as the number and density of positive monomeric species (Lin et al. 2008). The model also indicates the significance \((p < 0.05)\) of alkalinity in turbidity removal. This suggests that alkalinity plays significant contribution in the coagulation–flocculation process.

Equation (3) indicates a significant \((p < 0.05)\) turbidity response model equation. The equation depicts positive relationship between the linear effects (pH and alkalinity), the interactive effect (pH X alum dose, alum dose X alkalinity) and the quadratic effects ((pH)^2, (alum dose)^2) of the variable factors. The linear, interactive and quadratic effects have greater impact on turbidity removal. There is a correlation between turbidity removal and the coagulation optimization determinants. This assertion agrees with studies conducted by Pernitsky and Edzwald (2006). The pH, alum dosage and alkalinity values of 7.0 pH units, 80 mg/L and 96 mg/L as CaCO_3 provide significant impetus for sweep flocculation process. Turbidity removal enhanced by precipitated hydroxide molecule from alkaline also facilitates effective coagulation.

Equation (4) is colour removal model; the effects of the factors were similar to the removal of turbidity with the linear (pH), interactive (pH-alkalinity) and quadratic effects (pH^2 and alum dose^2) to be significant. The significance of the interactive effect of pH and alkalinity plays an essential role in colour removal at a lower dose at values 7.0 pH units, 63.2 mg/L coagulant and 80 mg/L as CaCO_3 provide significant impetus for sweep flocculation process. Turbidity removal enhanced by precipitated hydroxide molecule from alkaline also facilitates effective coagulation.

Equation (5) presents a good model for residual aluminium response. As indicated by the equation, the linear model effects of pH and alkalinity for the coagulation solution are substantial, with p values of 0.013 and 0.038, respectively. The interactive effects of pH and alum dosage and alum dosage and alkalinity on residual aluminium removal are significant with p values of 0.030 and 0.032, respectively. The quadratic effects of alum dose were significant \((p < 0.05)\). The residual aluminium response is a function of the amount of aluminum dose in the solution and depends on the conditions provided by pH and alkalinity (Yukselen and Gregory 2004). Here, more than one mechanism is employed to remove residual aluminium.

Equation (6) shows that phenanthrene removal is impacted significantly \((p < 0.05)\) by the linear effect of pH. However, the interactive effect of the factors was less significant (Table 5). Phenanthrene removal is related to pH effects (Trinh and Kang 2011; Hurst et al. 2004). Phenanthrene colloids by their nature make it possible to be coagulated due to their high molecular weight and hydrophobic character (Yukselen and Gregory 2004). They are not considered part of true water-soluble fraction and can be removed from the water by the application of charge neutralization (Yukselen and Gregory 2004). Solution pH that influences phenanthrene colloidal charges contributes significantly to the removal by charge neutralization.

\[
\text{Turbidity} = \frac{118.08 - 36.079 A + 0.029 C}{0.024 A + B - 0.001 B + C - 2.683 A^2 - 0.002 B^2} \quad (3)
\]

\[
\text{Color} = \frac{-57.855 + 17.127 A + 0.035 A + C - 1.444 A^2 - 0.002 B^2}{0.002} \quad (4)
\]

\[
\text{Aluminium. Res} = \frac{0.379 + 0.159 A + 0.002 A + B}{0.003} \quad (5)
\]

\[
\text{Phenanthrene} = \frac{48.448 - 9.198 A + 0.003 C^2}{0.003} \quad (6)
\]
solution alkalinity was 90.0 mg/L. Likewise, Fig. 3c indicates predicted aluminium residual of 0.0084 mg/L at alum dose of 63.18 mg/L at a pH range of 7.0. Figure 3d shows the phenanthrene removal plot. Removal occurs at pH 6.16 with value 1.6395 µg/L when the optimum dose and alkalinity were 80.0 mg/L and 80 mg/L, respectively. The removal efficiency reduces when moving away from the predicted values in either decreasing or increasing for the factors, resulting in reduced response value. Finally, optimum pH for colour, aluminium and phenanthrene removal was lower than that of turbidity removal. Turbidity removal occurring at higher pH > 6.5 has been studied by the Letterman and American Water Works Association 1999; Duan and Gregory 2003; Kim et al. 2001). The study shows that at this condition removal is due to sweep flocculation mechanism. Alternatively, the decrease in pH organic protonation and process reduction of charge density occurs and causes self-aggregation of anionic organic molecules, thereby leading to less coagulant to destabilize them. The process of complexation under low-pH conditions occurs simultaneously in the presence of aluminium hydroxide species (Bell-Ajy et al. 2000).

Table 7 shows targets and ranges for conditions and responses. The targets for pH, alum dose and alkalinity were 6.9 pH units, 71 mg/L and 80 mg/L as CaCO₃ with ranges 6.5–7.5 pH units, 70–90 mg/L and 70–90 mg/L, respectively. The response targets for turbidity, colour, residual aluminium and phenanthrene were 1.211NTU, 2.1272Pt.C0, 0.013 mg/L and 2.245ug/L. Setting the
optimization of the coagulation process at the required optimum targets, the boundary requirements and response outcome would be achieved. The optimum response is achieved at the set ranges by the application of desirability function. Notably, the range and target sets gave the best removal efficiency, supporting the individual 3D response plot. The desirability application can optimize all the conditions to achieve a more efficient response.

Figure 4a and b shows the 2D contour and overlay plot for all responses’ desired conditions. Individual responses and their optimization were achieved under different optimal conditions. Figure 4a shows a compromise between the optimum conditions for the four responses (turbidity, colour, residual aluminium and phenanthrene). Achieving response optimization occurs at pH 6.812; alum dosage 71.591 and alkalinity 90.000 mg/L given the desirability of 0.939.
Response outcome generated for turbidity, colour, residual aluminium and phenanthrene shows values of 1.211 NTU, 1.868 Pt.Co, 0.012 mg/L and 2.2265 µg/L, respectively.

Figure 4b is an overlay plot obtained at a defined limit of coagulation conditions; pH between 6.5 and 7.2, the dosage from 66.0 to 72.0 mg/L and alkalinity from 70.0 to 90.0 mg/L to optimize the response. The graphical optimization illustrates practical response values in the factor space and the region that fits the optimization criteria. Therefore, the overlay plot’s shaded area shows removal efficiency of 84.3% turbidity, 97.3% colour, 86.2% residual aluminium and 52.4% phenanthrene, respectively. Critical observation of the overlay plot indicates that the shaded area has a compromise value of 1.211 NTU, 1.870 Pt.Co, 0.012 mg/L and 2.265 µg/L for turbidity, colour, aluminium and phenanthrene residuals. The residuals were achieved at a pH of 6.81 and alum dosage 71.59 mg/L at an alkalinity of 90.0 mg/L.

Table 8 indicates the selected solution out of 33 optimized solutions found for the predicted model. A desirability value of 0.85235 shows a highly predictive model indicating optimum conditions for removing the four responses simultaneously. Desirability greater than 0.5 illustrates that the removal efficiency under the optimized conditions through coagulation–floculation is significant ($p < 0.05$) to optimize the responses with residual values of 1.369 NTU, 2.169 Pt.Co, 0.022 mg/L and 2.376 µg/L.

Table 9 tests the model equations’ adequacy for predicting the response values by comparing the experimental and predicted values at optimum conditions. Table 9 shows calibrated results obtained experimentally are close to the model-estimated response values for turbidity 1.283 NTU, colour, 2.427 Pt.Co, Residual aluminium, 0.022 mg/L and pH phenanthrene 2.263 µg/L. An insignificant margin of error below 0.027(%) as obtained between the experimental and predicted values indicates closeness between the observed and predicted values under the optimum conditions confirming the corresponding model’s suitability. The high correlation coefficient between the practical and predicted values in the testing domain also guarantees the model’s adequacy. The RSM approach used in this study was appropriate for
determining the optimum condition of coagulation and maximizing the response outcome of removal.

Conclusion

The study confirmed that optimum desirable coagulation conditions for the removal of the individual responses are different. The removal of turbidity, colour, residual aluminium and phenanthrene are significantly influenced by concentration and levels of pH, alum and alkalinity, which are the key coagulation conditions. The quadratic model shows highly significant polynomial model with a high correlation which was confirmed by insignificant lack of fit.

Optimization of coagulation conditions was possible to maximize the removal response outcome under a targeted desirability factor. The adequacy of the model was tested and had a margin of error less than 0.026% indicating a good agreement between experimental and predicted values under optimum desired conditions.

The study demonstrated that RSM can be used to successfully optimize coagulation–floculation processes in drinking water treatment. The methodology is economical and appropriate to optimize the coagulation conditions to maximize the coagulation–floculation process' response outcome.

Author contributions HM-A conceptualized the research topic and formulated the objectives and methodology of the research. KBN carried out both the laboratory and field investigation as well as the data curation of the research work. RB carried out laboratory work and also assisted in the writing of the manuscript. SW is the corresponding author and participated in the writing of the manuscript (original draft preparation) and reviewed and edited the manuscript.

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Declarations

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Ethical conduct This manuscript has never been published in any journal or sent to any journal for consideration and publication. Strict scientific ethical standard was adhered to.

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