



## Response Surface Methodological Approach to Optimize the Process Parameters Using PFSiS - A hybrid Coagulant in Water Treatment

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### Abstract

*Polyferric silicate sulphate (PFSiS), a hybrid coagulant was amalgamated by co-polymerization technique using Ferric salt and Polysilicic acid at different OH/Fe ratio and Fe/Si molar ratios. The main intention of this paper was to optimize the factors for the turbidity removal in coagulation-flocculation process in water treatment using Box Behnken method. Effectiveness of important process parameters Fe/Si molar ratio, OH/Fe molar ratio, pH and dosage were enumerated, optimized and designed effectively. Valid quadratic polynomial models were obtained. Hence, PFSiS has proven the capability for the turbidity removal in low turbid water.*

**Keywords:** Coagulation, Flocculation, Turbidity, Polyferric silicate sulphate, Response surface methodology.

### Introduction

The quality of water is directly associated to the quality of our lives due to its increased demand and also the decrease in resources and hence becomes requisite for the critical needs of society. Water qualities enmesh both health and aesthetic concerns depending on the purpose of utilization, consumption and also the contaminants present. Therefore, the purpose of water treatment is to accomplish safe guarded and aesthetically pleasing water. This mainly focuses on water that free from harmful substances such as chemicals, microbes, as well as acceptable taste and odour.

Suspended and colloidal matter present in water and waste water result in turbidity. Coagulation and flocculation processes are foremost parts of water treatment regarding the elimination of colloidal and suspended particles and they are used together to remove turbidity that impart colour to water source and retain microorganisms.

Usually colloidal particles typically have larger surface area and negatively charged. The chemical which is added to water with the intention of destabilizing the negatively charged particles that are colloidal in nature is called coagulant. Hybrid materials used in coagulation/flocculation of water are components obtained from the addition of effective composition to the original constituent to strengthen the aggregating power. The potentiality of polymerized metal species in Inorganic Polymeric Flocculants developed them to perform more efficiently than regular coagulants such as alum and ferric chloride.

The fusion of silica and ferric salt advance the constancy of hydrolyzed ferric salt and the establishment of large flocs<sup>1,2</sup>.

PFSiS has OH/Fe molar ratio of 0.2, 0.4, 0.6 and Fe/Si molar ratio of 1, 2 and 3<sup>3,4</sup>. The classical optimization technique is time consuming and expensive. Hence, Statistical experimental design is a suitable technique to acquire beneficial and analytically valid models of phenomenon by executing least number of experiments<sup>5</sup>.

Response surface methodology (RSM) design allow to estimate interaction and quadratic effects and one of the common designs under RSM is Box Behnken method<sup>6-8</sup>.

The main intention of this study was to evaluate the optimal condition for turbidity removal in water treatment. The experimental runs in deal with Box Behnken method in Response Surface Methodology is used for the entire study.

### Materials and Methods

**Synthetic Turbid Water:** In this study synthetic water is used to maintain a consistent water quality while conducting the experiments. Synthetic turbid water for the jar tests was prepared by using kaolin clay materials to water. About 15 g of the kaolin clay materials was added to 500 ml of distilled water and the suspension was mixed for about 1 hour to effect a homogenous distribution of clay particles. Then it was kept in an undisturbed condition for at least one day for full hydration of the clay materials to achieve the desired turbidity just before coagulation<sup>9</sup>.

**Preparation of Coagulant:** Polysilicic acid constitutes rapid mixing of 23.5 ml of 1.5M hydrochloric acid and 50 ml of 0.5 mol/L silicon di oxide solution at decreased pH 2 to yield 0.3M Psi solution. Also, Ferric solution was prepared from Ferric sulfate (Iron III sulfate) then the basic solution Sodium

hydroxide is initially added to the Ferric solution to produce the desired OH/Fe molar ratio and appropriate amount of polysilicic acid is introduced to attain the preferred Fe/Si molar ratio. Final volume was maintained to 100ml by adding distilled water.

Thus obtained final volume of 100ml in each beaker contained the varying Fe/Si and OH/Fe molar ratio with constant ferric concentration as given in Table-1. This process of preparation of coagulant by polymerization is called co-polymerization technique<sup>10,11</sup>.

**Coagulation Method:** Coagulation- flocculation process is conducted using jar test apparatus and synthetically prepared turbid water samples with initial turbidity 44 NTU. The experiment was run in accordance with Box Behnken method.

Percentage removal of turbidity was calculated by using the successive formula,

$$\text{Percentage removal} = (T2 - T1) / T1 * 100$$

Where, T1 –Initial turbidity and T2 –Final turbidity.

**Experimental design and Statistical analysis:** The chief intent is to optimize the response surface that is controlled by various process parameters<sup>8</sup>. Design expert software is used for summarizing design, analytical modeling and optimization of each variable. The independent variables used in this study are Fe/Si molar ratio (A), OH/Fe molar ratio (B), pH (C) and dosage in terms of mg/l (D) each with three levels. Turbidity removal efficiency (y) was taken as dependent variable.

**Table-1**  
**Preparation of PFSiS having assorted OH/Fe and Fe/Si molar ratios**

Coagulant No.	Volume of Ferric solution (ml)	Volume of 0.5M NaOH (ml)	Volume of 0.3M Psi solution (ml)	Final volume	Fe/Si molar ratio	OH/Fe molar ratio
1	30	3	25	100	1	0.2
2	30	6	25	100	1	0.4
3	30	9	25	100	1	0.6
4	30	3	12.5	100	2	0.2
5	30	6	12.5	100	2	0.4
6	30	9	12.5	100	2	0.6
7	30	3	8.3	100	3	0.2
8	30	6	8.3	100	3	0.4
9	30	9	8.3	100	3	0.6

**Table-2**  
**Levels of Factor**

Factor	Level 1	Level 2	Level 3
A	0.2	0.4	0.6
B	1	2	3
C	5	7	9
D	0.5	1	1.5

**Table-3**  
**Experimental Runs**

Run No.	Fe/Si	OH/Fe	pH	Dosage (mg/l)
1	2	0.6	7	1.5
2	2	0.4	7	1
3	2	0.2	9	1
4	1	0.4	9	1
5	1	0.4	7	0.5
6	2	0.4	9	0.5
7	2	0.6	7	0.5
8	2	0.4	9	1.5
9	2	0.2	7	1.5
10	2	0.2	7	0.5
11	2	0.4	7	1
12	1	0.6	7	1
13	2	0.6	5	1
14	2	0.2	5	1
15	3	0.4	9	1
16	3	0.6	7	1
17	1	0.2	7	1
18	3	0.4	5	1
19	2	0.4	7	1
20	2	0.4	5	0.5
21	1	0.4	7	1.5
22	2	0.4	7	1
23	1	0.4	5	1
24	2	0.4	5	1.5
25	3	0.4	7	1.5
26	3	0.4	7	0.5
27	2	0.4	7	1
28	3	0.2	7	1
29	2	0.6	9	1

## Results and Discussion

Analysis of variance was used for analyses of the data. The coefficient of determination R<sup>2</sup> shows the quality of fit polynomial model and its F test shows the statistical significance. The P value with 95% confidence level checks the model terms. Three dimensional plots and their corresponding contour plots were determined based on the effects of four factors at three levels. Equation (1) presents the models for turbidity removal efficiency (%). The quadratic model statistical results for turbidity removal were summarized as follows:

**Table-4**  
**Quadratic model statistical results**

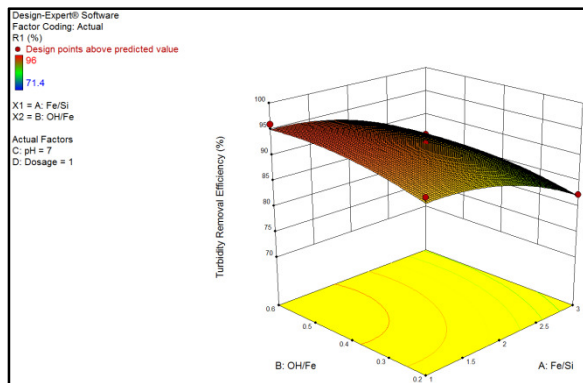
Std. Dev.	1.28
Mean	84.65
R-Squared	0.9838
Adj R-Squared	0.9675
Pred R-Squared	0.9065
C.V. %	1.5
PRESS	131.09
Adeq Precision	24.760

Table-2 and Table-3 shows the levels of each variable and the experimental runs respectively. The “Pred R-Squared” of 0.9065 is in rational agreement with the “Adj R-Squared” of 0.9675. “Adeq Precision” fix the signal to noise ratio and was found to be 24.760, which indicates an adequate signal. A ratio greater than 4 is desirable. This model can be used to navigate the design space.

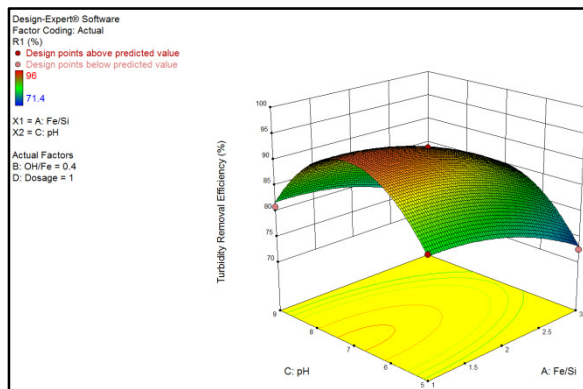
**Three dimensional plots:** The response surface plots retrieved from the Design Expert software give a 3D view of the turbidity removal efficiency with different combination of independent variable. Some of the interaction effects were shown in Figure. The relationship between the turbidity removal efficiency and these four factors are shown in Figure 1-6 for low turbid water. Each plot shows the effects of two variables within their studied ranges, with the other variable fixed to zero level. The response surface better visualize the tendency of each factor to influence the turbidity removal efficiency. The shape of the contour plot shows the natures and extents of the interactions between factors.

**Optimization and validation experiment:** Optimized condition under stated constraints were achieved for highest suitability at Fe/Si molar ratio 1.68, OH/Fe molar ratio 0.54, pH 6.97 and dosage 1.34 mg/l. Under this condition, 96.03% turbidity removal was predicted depending on desirability

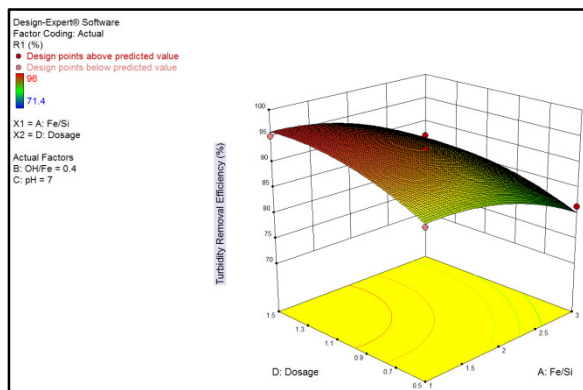
function of 1.00. After confirm the accuracy of the predicted model and consistency of optimum combination, an additional experiment was carried out at optimum condition. The experimental value 99.1% was found to agree well with predicted 98.44%. The low error in the experimental and predicted value shows good agreement of the results obtained from models and experiments. These results confirm that RSM is a competent tool for optimizing the operational condition for turbidity removal in coagulation.



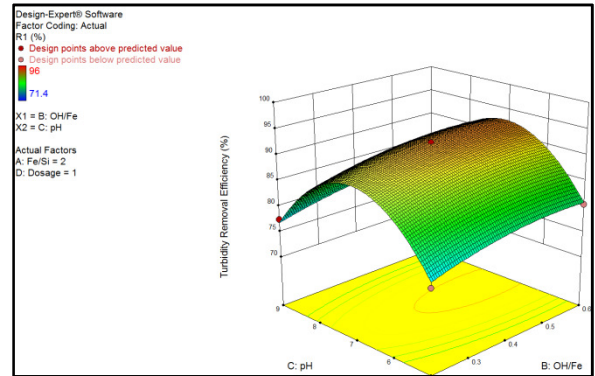
**Figure-1**  
 Interaction between Fe/Si and OH/Fe



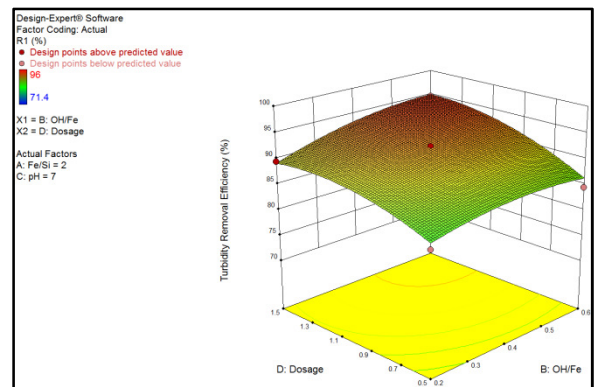
**Figure-2**  
 Interaction between Fe/Si and pH



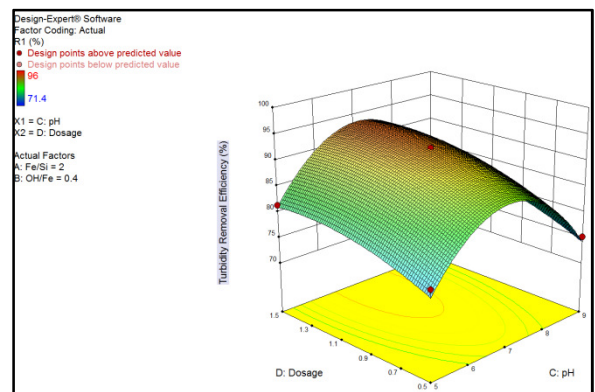
**Figure-3**  
 Interaction between Fe/Si and Dosage



**Figure-4**  
 Interaction between OH/Fe and pH



**Figure-5**  
 Interaction between OH/Fe and Dosage



**Figure-6**  
 Interaction between pH and Dosage

## Conclusion

Polyferric Silicate Sulphate (PFSiS) acts as a good coagulant in water treatment and highly suitable for low turbid water. Box-Behnken method (BBM) of Response Surface Methodology (RSM) can be used to determine the importance of each of studied factors in Coagulation Flocculation process. The turbidity removal efficiency decreases with rise in Fe/Si molar ratio. The turbidity removal efficiency increases with rise in

OH/Fe molar ratio and dosage. The turbidity removal efficiency rises till the pH reaches neutral (pH 7). Agreement of the quadratic model with the experimental data was reasonable. Hence PFSiS can be the potential coagulant for the water treatment.

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