

# Optimization of the Operating Conditions of Turbidity Removal from Synthesized Dairy Wastewater Using Pumpkin Seed as a Coagulant

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**Abstract** - This work has been carried out to apply response surface methodology of the Design Expert to obtain the optimum conditions for using pumpkin seed coagulant to treat dairy wastewater using central composite design. The wastewater used for the research was synthesized by dissolving instant powdered milk in tap water. Also, the coagulant used for the treatment of the wastewater was prepared by processing the pumpkin seed obtained from Muda Lawal Market of Bauchi, Bauchi State, Nigeria. Before applying the Design Expert, jar test was first carried out to determine the optimum coagulant dose for the treatment of the synthesized wastewater as 4 g/l. Thereafter, twenty experiments were designed and performed according to the central composite design of response surface methodology of the Design Expert. The results of the experiments, together with the factors used, were modelled, analysed, modified and optimized to obtain the optimum values for the wastewater treatment. The analyses of variance of the full quadratic model developed revealed that it was significant with a p-value less than 0.0001. However, the model was found to contain some insignificant factors, which made the model to undergo modification. The results obtained from the simulation of the modified quadratic model developed showed that it was very good and capable of predicting the behaviour of the process well because its R-Squared, Adj R-Squared and Pred R-Squared values were estimated to be 0.9281, 0.8861 and 0.6693, respectively. Also, the results obtained from the experiments carried out with the design of the response surface methodology showed that the minimum turbidity value given by response surface methodology was better than that of the jar test. Therefore, response surface methodology has been successfully applied to obtain the optimum conditions required for removing turbidity from dairy wastewater using pumpkin seed coagulant.

**Keywords:** Dairy wastewater, coagulant, pumpkin seed, response surface methodology, Design Expert.

## 1 INTRODUCTION

Wastewater from industries varies so greatly in both flow and pollution strength. It is impossible to assign fixed values to their constituents. Industrial wastewaters may contain suspended, colloidal and dissolved (mineral and organic) solids. In addition, they may be either excessively acidic or alkaline and may contain high or low concentrations of coloured matter. These wastewaters may contain inert, organic or toxic materials and possibly pathogenic bacteria (Alturkman, 2006). As such, they must be treated or processed to make their concentrations be within acceptable limit before being released into the environment or water bodies. Treatment of these wastewater types is an important issue in environmental protection as the wastewater normally contains pollutants that, if not efficiently treated, can cause serious hazards to the environment.

The food processing industry seeks cost-effective reduction and recycling technologies for food processing wastewaters due to the enforcement of wastewater discharge regulations and escalating sewage surcharges. These technologies include both source reduction options (technologies to reduce the amount of water used) and treatment options (technologies to reduce the amount or contamination level of wastewaters requiring discharge) (Philips, 1997).

Over the last few decades, several methods have been applied to treatment of industrial wastewater; these include membrane separation, solvent extraction, chemical reduction, chemical precipitation, evaporation, lime coagulation, cementation, ion exchange, reverse osmosis and electrodeposition (Singh and Kaushal, 2013). However, of all these methods, the commonly used ones are pre-liming, coagulation, flocculation, sedimentation and filtration.

The production of potable water from most raw water sources usually entails the use of a coagulation/flocculation stage to remove turbidity in the form of suspended and colloidal materials. This process plays a major role in industrial wastewater treatment by reducing turbidity, bacteria, algae, colour, organic compounds, and clay particles. The presence of suspended particles could clog filters or impair disinfection process, thereby dramatically maximizing the risk of waterborne diseases. Many coagulants, one of which is alum, are widely used in conventional water treatment processes, based on their chemical characteristics. These coagulants are classified into inorganic, synthetic organic polymers, and natural coagulants (Vara, 2012).

Alum has been the most widely used coagulant because of its proven performance, cost effectiveness, relatively easy handling and availability. Recently, much attention has been drawn to the extensive use of alum. Aluminium, which is contained in alum, is regarded as an important poisoning factor in dialysis encephalopathy. It (aluminium) is one of the factors which might contribute to Alzheimer's disease. Some synthetic organic polymers such as acrylamide have neurotoxicity and strong carcinogenic effect. Besides, the use of alum salts is inappropriate in some developing countries because of the high costs of imported chemicals and low availability of chemical coagulants (Vara, 2012). It, therefore, means that there is the need to embark on the use of natural coagulants for water

treatment because the use of chemical coagulants such as aluminium salt to treat industrial wastewater has been found to be harmful to human health.

Furthermore, a number of studies have pointed out that the introduction of natural coagulants as substitutes for metal salts may ease the problems associated with chemical coagulants. Natural macromolecular coagulants are promising and have attracted the attention of many researchers because of their abundant source, low price, multi-purposeless, and biodegradation. Okra, rice, and chitosan are natural compounds which have been used in turbidity removal. The extracts of the seeds have been mentioned for drastically reducing the amount of sludge and bacteria in sewage (Vara, 2012). Other used natural coagulants include *Nirmali* seed and maize, mesquite bean and *Cactus latifari*, *Moringa oleifera* seed, and pumpkin seed. The main advantages of using plant-based coagulants as water treatment materials are apparent; they are cost-effective, unlikely to produce treated water with extreme pH and highly biodegradable. Naturally occurring coagulants, such as pumpkin seed, are usually presumed safe for human health (Birima et al, 2013).

Pumpkin (the seed of which is shown in Figure 1) is one of the most delicious vegetables. It is commonly used in cooking various dishes. This pulpy vegetable has many seeds that are often thrown away as most of us are unaware of their wonderful taste and health benefits (Reddy, 2014).



Figure 1: Pumpkin seed (*Pepitas, Cucurbita pepo*) (<http://www.whfoods.com/genpage.php?tname=foodspice&dbid=82>)

Pumpkin seeds also have water soluble proteins that are released from the powdered seed that attach themselves and bind between the suspended particles and, thereby, forming larger agglomerated solids. These flocculated solids would be allowed to settle prior to boiling and subsequent consumption of the water. The active fraction of the seeds has received much interest. Investigations by professor Tauscher at the University of Karlsruhe, Germany have revealed that the coagulant properties of the seeds are due to a series of low molecular weight cationic proteins. The potential toxicity of the seeds has been considered in two major studies; the conclusion of both were that the doses typically used for water treatment posed no serious threat to human health (Ali et al., 2010). As such, pumpkin seed can be used as a coagulant for the treatment of industrial wastewater because it is not harmful to the health, readily available, accessible and affordable.

Some researchers have worked on the use of natural coagulants for water treatment. Among them is the work of Ndabigengesere (1995) that studied the active agents and mechanism of coagulation of turbid waters using *Moringa oleifera*. He was able to discover that the active agent in *Moringa oleifera* extracts were diametric cationic proteins, and that the mechanism of coagulant with *Moringa oleifera* appeared to consist of adsorption and neutralization of the colloidal charges compared to alum. Also, Diaz et al. (1999) carried out a

preliminary evaluation of turbidity removal by some natural coagulants (*Cactus latifaria* and seeds of *Prosopis juliflora*) and found out that both *Cactus latifaria* and seeds of *Prosopis juliflora* were effective in reducing the pollutant of the water they considered to the required standard of 5 NTU from both high (100-200 NTU) and low (30-40 NTU) turbidity ranges.

Generally, the factors affecting the coagulation process of pumpkin seed include coagulant dose, coagulation time and pH. These factors affect the manner in which the seed is used to treat water. Therefore, it is necessary to obtain their values that will treat water to its best. In other words, the optimum parameters of the factors need to be obtained and used to, properly, carry out water treatment. One of the methods obtained from the literature that can be used for optimizing a process like is the response surface methodology.

Response surface methodology (RSM) is a widely used technology for rational experimental design and process optimization in the absence of mechanistic information (Box and Draper, 1987; Myers and Montgomery, 1995; Giwa and Giwa, 2012). RSM initiates from Design of Experiments (DoE) to determine the values of the factors to be used for conducting experiments and collecting data. The data are then used to develop an empirical model that relates the process response to the factors. Subsequently, the model facilitates to search for better process response, which is

validated through experiment(s). The above procedure iterates until an optimal process is identified (Chi et al., 2012; Giwa and Giwa, 2012). RSM has seen diverse applications in almost every area of scientific research and engineering practice, including the development of chemical and biochemical processes (Agatonovic-Kustrin et al., 1998; Baumes et al., 2004; Dutta et al., 2004; Hadjmohammadi and Kamel, 2008; Shao et al., 2007; Tang et al., 2010; Yan et al., 2011a,b; Giwa and Giwa, 2012). It is perceived that this good method of optimization can be extended to wastewater treatment.

Therefore, the aim of this work is to obtain the optimum conditions for treating synthesized dairy

wastewater in order to remove its turbidity using a coagulant developed from pumpkin seed by employing the central composite design of the response surface methodology of the Design Expert. The optimum values obtained using the response surface methodology will be compared to those obtained from the conventional jar experimental method.

## 2 METHODOLOGY

### 2.1 Coagulant Synthesis

Given in Figure 2 below is the flowchart of the steps involved in preparing the pumpkin seed coagulant used in this work.

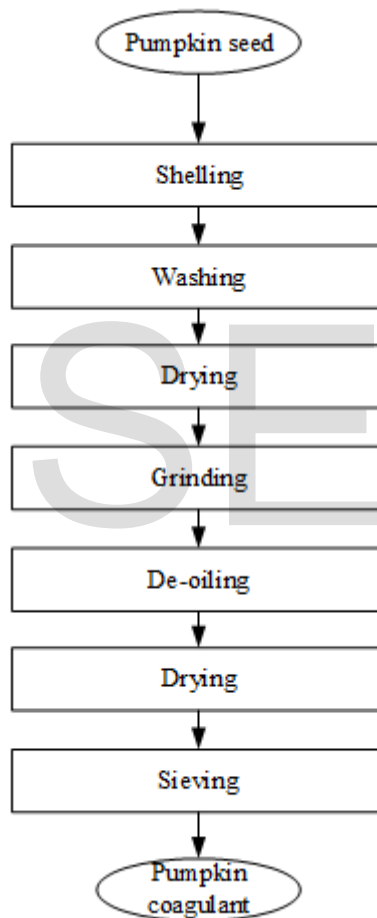


Figure 2: Flowchart for coagulant preparation





Figure 3: Soxhlet Extractor

In preparing the coagulant from pumpkin seed, moist seed was obtained from Muda Lawal Market in Bauchi, Bauchi State of Nigeria and shelled, washed and dried in an oven at a temperature of 50 °C for about 7 hrs. After that, the seed was grinded and placed in a 250 ml Soxhlet extractor (Figure 3) operated at 44 °C for 8 hr for its oil content to be extracted. During the oil

extraction operation, n-hexane was used as the solvent. The resulting cake, shown in Figure 4, obtained after drying the pumpkin seed powder from which oil had been extracted for 5 min at 50 °C and sieving yielded the powder used as the coagulant for the treatment of the dairy wastewater that was synthesized in this work.



Figure 4: Pumpkin seed cake

The synthetic dairy wastewater used in this research work was prepared by dissolving instant powdered milk in tap water. The solution was thoroughly shaken and left at room temperature in a tightly covered container for about 24 hr.

The turbidity of the synthesized dairy wastewater was measured before and after the treatment (experiment) using DR/890 Colorimeter. A pH meter was also used to measure the pH of the synthesized wastewater. Prior to each experiment, pH adjustment was done by adding 0.5 M H<sub>2</sub>SO<sub>4</sub> or NaOH, as the case demanded, to the wastewater.

### 2.2 Jar Test

A jar test apparatus (Flocculator SW6) with six beakers was used to find the optimum coagulant dose that would give the minimum residual turbidity or the maximum turbidity removal. In order to achieve this, 500 ml of the synthesized dairy wastewater was poured into each of the beakers labelled from 1-6 and coagulant dose was varied between 1 and 6 g/l. The coagulation was carried out for 15 minutes out of which the first 5

minutes were used for rapid mixing at a speed of 150 rpm, while at the last 10 min of the coagulation, the mixture was moderately mixed at 80 rpm. After the coagulation process, the mixture was allowed to settle for 30 minutes and filtered to obtain the treated wastewater.

### 2.3 Experimental Design

In this aspect of the work, with the aid of Design Expert 7.0.0 (Stat-Ease, 2005), a set of 20 experiments, as shown in Table 3, was generated according to central composite design of the response surface methodology using alpha value of 1.6818. The factors and their actual and coded values are as given in Table 1. The experimental results obtained after the experiments were analysed using a full quadratic model that was later modified to improve the significance and the prediction capability of the developed model. The response (residual turbidity) was then minimized using the numerical optimization approach of Design Expert to obtain the optimum operating conditions of the dairy wastewater treatment process.

Table 1: The ranges of the experimental factors

Factors	Symbols	-α	-1	0	+1	+α
pH	A	5.5	6.11	7	7.89	8.5
Coagulant dose, g/l	B	1.0	1.81	3.0	4.19	5.0
Coagulation time, min	C	5	6.01	7.5	8.99	10

## 3 RESULT AND DISCUSSION

The wastewater used in this work was synthesized, and after the synthesis, it was analysed to get its initial characteristics. It was obtained from the initial analyses of the synthesized wastewater that its pH was 6.84 while its turbidity was 467 NTU. Even though the pH of the wastewater was approximately equal to that of the water that could be discharged into the water body directly, its turbidity was found not to be friendly. This was what, actually, necessitated the need for the treatment. The first treatment (jar test) given to the wastewater was carried out by varying the coagulant dose in order to see its effects on the removal of the turbidity of the water.

Shown in Table 2 are the results obtained from the jar experiments carried out in which the pumpkin seed coagulant dose was varied while the other factors (pH and coagulation time) were kept constant, in order

to determine the dose that could give the lowest turbidity of the water being considered. From the results, it was observed that increase in coagulant dose led to a corresponding decrease in turbidity, and the optimum dose of the coagulant was observed to be 4 g/l because that was the dose that was used to achieve a minimum turbidity of 135 NTU. An increase in the coagulant dose after that optimum dose of 4 g/l was found to result in an increase in the turbidity of the water. Under the optimum conditions (that is, pH of 6.84, coagulation time of 15 min, and coagulant dose of 4.0 g/l), the final turbidity percentage removal was calculated to be 71.09%. This value of the percentage turbidity removal was found not be too bad, but it was desired to improve it a bit. As such, a better statistical method of optimization, known as response surface methodology, which varies all the parameters to obtain the optimum values was employed.

Table 2: Jar experimental results

Run	pH	Coagulant dose (g/l)	Coagulation time (min)	Residual turbidity (NTU)
1	6.84	1.0	15	290
2	6.84	2.0	15	286
3	6.84	3.0	15	215
4	6.84	4.0	15	135
5	6.84	5.0	15	224
6	6.84	6.0	15	316

The results of the experiments designed, with the aid of response surface methodology, and using the central composite design, and carried out for testing the coagulation and water treatment capability of the pumpkin seed coagulant developed are given in Table 3. According to the table, the turbidity removal of the pumpkin seed coagulant was found to be affected by the variations in the input factors (pH, coagulation time and coagulant dose). For instance, the experiment carried out at minimum values of all the factors (pH of 6.11, coagulant dose of 1.81 g/l and coagulation time of 6.01 min) was found to yield water with turbidity of 273 NTU (Run 19) while the one performed at the maximum values of the factors (pH, coagulant dose and coagulation time of 7.89, 4.19 mg/l, 8.99 min,

respectively) gave water with residual turbidity of 194 NTU (Run 9). This implies that as the factors of the experiments were increased from minimum to maximum values, there was a decrease in the turbidity and, thus, an increase in the percentage turbidity removal. Also, at the centre point of the experiments (that is, pH = 7, coagulant dose = 3 g/l, coagulation time = 7.5 min), the average residual turbidity was found to have a value of 252 NTU (runs 20, 2, 16, 8, 7, 11). It has, thus, been found that the average residual turbidity value obtained from the centre point experiments was less than that obtained at the minimum points but higher than that produced by the maximum values of the factors used in the experiment.

Table 3: Experimental factors and responses

Run	Factors			Response	Turbidity removal, %
	A:pH	B:Coagulant dose, g/l	C:Coagulation time, min	Residual turbidity, NTU	
1	5.50	3.00	7.50	224	52.0343
2	7.00	3.00	7.50	250	46.4668
3	7.00	3.00	5.00	192	58.8865
4	7.00	5.00	7.50	132	71.7345
5	6.11	4.19	6.01	203	56.5310
6	6.11	4.19	8.99	163	65.0964
7	7.00	3.00	7.50	250	46.4668
8	7.00	3.00	7.50	256	45.1820
9	7.89	4.19	8.99	194	58.4582
10	8.50	3.00	7.50	232	50.3212
11	7.00	3.00	7.50	256	45.1820
12	7.00	3.00	10.00	197	57.8158
13	7.89	1.81	8.99	367	21.4133
14	7.89	1.81	6.01	266	43.0407
15	6.11	1.81	8.99	237	49.2505
16	7.00	3.00	7.50	250	46.4668
17	7.89	4.19	6.01	184	60.5996
18	7.00	1.00	7.50	320	31.4775
19	6.11	1.81	6.01	273	41.5418
20	7.00	3.00	7.50	250	46.4668

Furthermore, increasing the pH of the wastewater (run 10) and coagulation time (run 12) beyond their normal maximum values for the experiment resulted in increase in residual turbidity. This was noticed when the pH was increased from negative alpha value (5.5) to positive alpha value (8.5) in which the residual turbidity, then, was found to increase from 224 NTU (run 1) to 232 NTU (run 10). Similarly, an increase in the coagulation time was discovered to lead to increase in residual turbidity from 192 NTU (run 3) to 197 NTU (run 12).

Within the ranges of the factors considered in the axial experiments, the coagulant dose was found to be the only factor that seriously affected the turbidity removal of the pumpkin seed coagulant. For instance,

$$Y = 114.49 - 12.73A + 117.33B + 5.53C - 13.08AB + 7.63AC - 6.72BC - 4.86A^2 - 3.23B^2 - 7.11C^2 \quad (1)$$

The developed model, which was quadratic in nature, was analysed, and the results of the analysis are as given in Table 4.

Table 4: Results of analysis of surface response full quadratic model of the wastewater treatment process

Source	Sum of squares	Degree of freedom	Mean square	F value	p-value	
Model	49993.82	9	5554.87	16.5	< 0.0001	significant
A-pH	1613.75	1	1613.75	4.79	0.0534	
B-Coagulant Dose	37452.13	1	37452.13	111.22	< 0.0001	
C-Coagulation Time	137.98	1	137.98	0.41	0.5365	
AB	1540.12	1	1540.12	4.57	0.0582	
AC	4371.13	1	4371.13	12.98	0.0048	
BC	1128.12	1	1128.12	3.35	0.0971	
A <sup>2</sup>	215.57	1	215.57	0.64	0.4422	
B <sup>2</sup>	301.61	1	301.61	0.9	0.3663	
C <sup>2</sup>	3557.53	1	3557.53	10.56	0.0087	
Residual	3367.38	10	336.74			
Lack of Fit	3319.38	5	663.88	69.15	0.0001	significant
Pure Error	48	5	9.6			
Cor Total	53361.2	19				
R-Squared = 0.9369;		Adj R-Squared = 0.8801;		Pred R-Squared = 0.4997		

As it can be observed from the table, even though the analysis of the model revealed that the entire model, having a p-value of 0.0001, was significant with R-squared, Adj R-squared and pred R-squared values of 0.9369, 0.8801 and 0.4997 respectively, it was discovered that some of the factors considered were insignificant because their p-values were greater than 5% (that is,

comparing runs 18 and 4, it was observed that increasing the coagulant dose from negative alpha value to positive value resulted in decrease in residual turbidity from 320 NTU to 132 NTU. The maximum turbidity removal, in this case, when the pH was 7.00 and coagulation time was 7.5 min, was obtained to be approximately 71.73%.

Using the response obtained from the experiments carried out and given in Table 3, a model relating the residual turbidity of the synthesized wastewater to the factors (pH, coagulant dose and coagulation time) affecting the turbidity removal of the pumpkin seed coagulant that were considered was developed to be:

with 95% confidence level). Besides, the low value of the pred R-squared of the model was an indication that the model might not be able to predict the behaviour of the process very well. As such, those insignificant factors, except the coagulation time that was retained to avoid hierarchy problem of the model, were removed from the model so that it (the model) could be modified and



improved. The modified model, which had its R-squared, Adj R-squared and pred R-squared values of 0.9281, 0.8861 and 0.6693 respectively was obtained to be:

$$Y = 394.97 - 80.80A + 97.92B + 0.36C - 13.08AB + 17.63AC - 6.72BC - 6.77C^2 \quad (2)$$

Table 5: Analysis of variance results for surface response modified quadratic model

Source	Sum of squares	Degree of freedom	Mean square	F value	p-value	
Model	49522.63	7	7074.66	22.12	< 0.0001	significant
A-pH	1613.75	1	1613.75	5.04	0.0443	
B- Coagulant Dose	37452.13	1	37452.13	117.08	< 0.0001	
C-Coagulation Time	137.98	1	137.98	0.43	0.5237	
AB	1540.12	1	1540.12	4.81	0.0486	
AC	4371.13	1	4371.13	13.66	0.0031	
BC	1128.12	1	1128.12	3.53	0.0849	
C <sup>2</sup>	3279.41	1	3279.41	10.25	0.0076	
Residual	3838.57	12	319.88			
Lack of Fit	3790.57	7	541.51	56.41	0.0002	significant
Pure Error	48	5	9.6			
Cor Total	53361.2	19				

R-Squared= 0.9281;      Adj R-Squared = 0.8861;      Pred R-Squared = 0.6693;

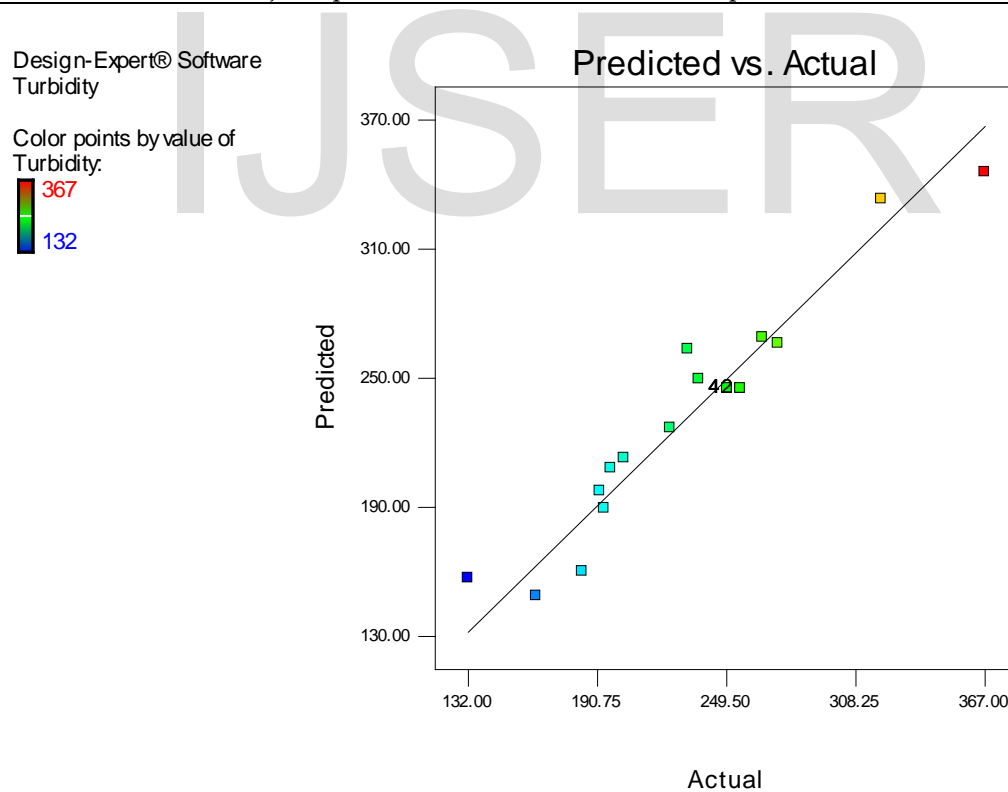


Figure 5: Predicted and experimentally measured residual turbidity of the synthesized dairy wastewater

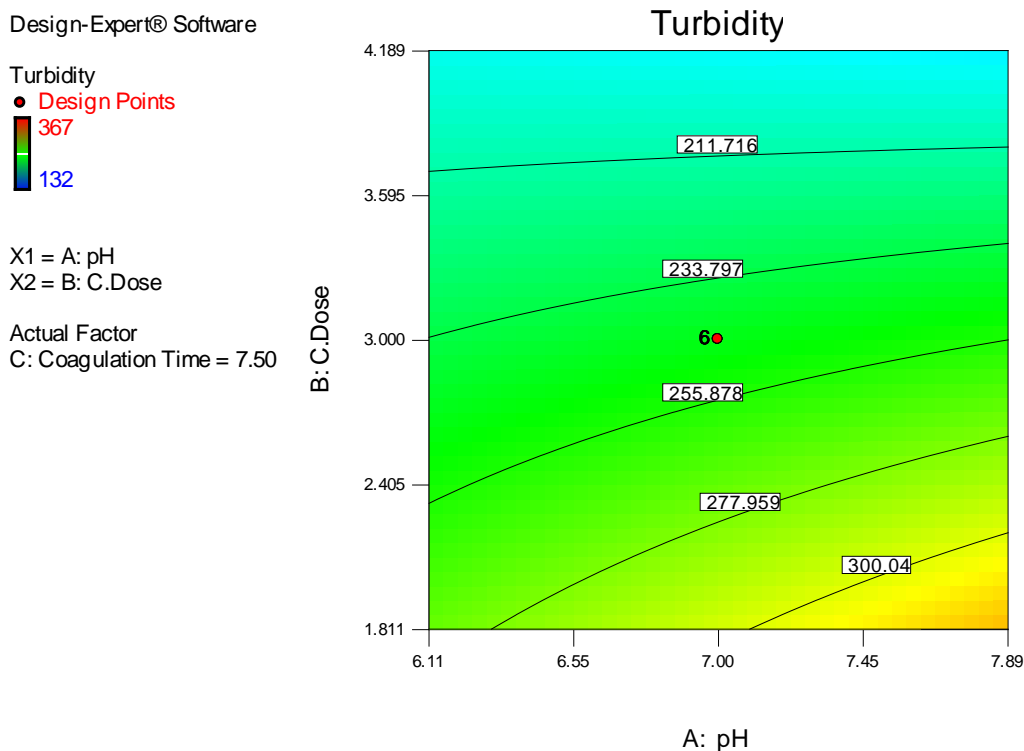


Figure 6. Effect of pH and coagulant dose on turbidity removal capacity of pumpkin seed coagulant

Moreover, the modified model was analysed and its results of analysis, given in Table 5, have revealed that it (the model) was not only significant but can also perform well in prediction because an improvement has been recorded in its pred R-squared value. It has, thus, been discovered that the modified model has a better capability of predicting the behaviour of the process because its pred R-squared has increased from 0.4997 to 0.6693.

After obtaining the modified model that was discovered to have good ability of predicting the behaviour of the process, it was simulated for prediction and the predicted residual turbidity values were plotted against the actual (experimental) values of the residual turbidity obtained from the treatment of the synthesized dairy wastewater using the developed pumpkin seed

coagulant, as shown in Figure 5. From the figure, it was discovered that good correlations were found to exist between the predicted and the actual residual turbidity values.

Also investigated in this work was the interaction occurring between the factors considered to be influencing the turbidity removal of the pumpkin seed coagulant from the wastewater. As a result of this, shown in Figure 6 is the interactive effect of pH and coagulant dose on the turbidity removal capacity of the pumpkin seed coagulant. According to the figure, increasing the coagulant dose while decreasing pH was found to lead to decrease in the residual turbidity of the wastewater, which indicated increase in the turbidity removal capacity of the coagulant.

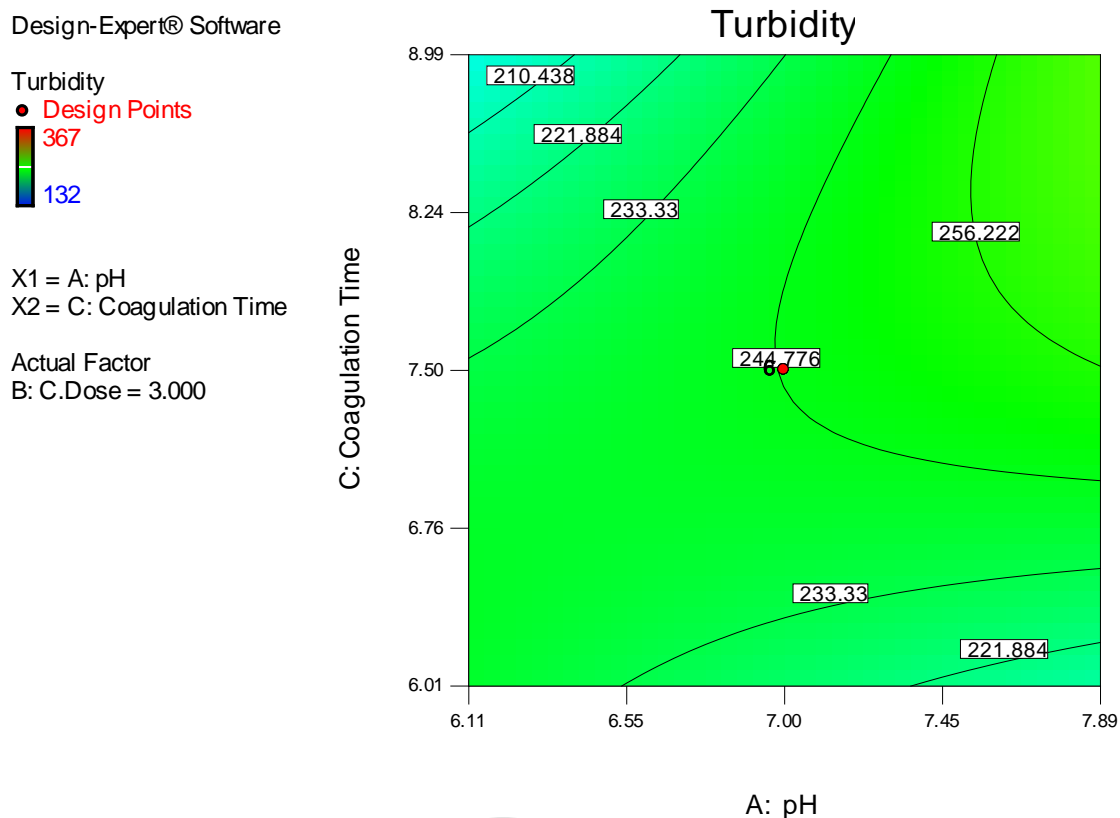


Figure 7. The effect of pH and coagulation time on turbidity removal capacity of pumpkin seed coagulant

Also given in Figure 7 is the interactive effect of pH and coagulation time on the turbidity removal capacity of the pumpkin seed coagulant. From the figure, it was noticed that, up to the centre point, a decrease in coagulation time with increase in pH was observed to lead to increase in residual turbidity. This means that the factors that are influencing the turbidity removal of the wastewater using the pumpkin seed coagulant must be regulated, especially, to optimum in order to achieve maximum efficiency of the treatment process.

Based on this, the modified model of the process was optimized, and the results obtained from the optimization carried out, with the aid of Design Expert, showed that the minimum residual turbidity that could be achieved was 148.63 NTU when the optimum values of pH, coagulant dose and coagulation time were 6.11, 4.19 g/l and 8.99 min, respectively.

Now, comparing the results obtained using jar experimental method and the response surface methodology, it was found that the minimum value of turbidity given by the jar experimental method was 135 NTU while that given by the response surface methodology was 132 NTU. It, therefore, implied that the response surface methodology employed in this

work was able to produce a better result than the conventional jar experimental method of optimizing turbidity removal capacity of coagulants.

#### 4 CONCLUSION

From the results obtained, it has been discovered that the central composite design of the response surface methodology of the Design Expert has been successfully applied to optimizing the use of pumpkin seed coagulant for the treatment of dairy wastewater. It was also found that the turbidity removal of the developed coagulant was largely affected by coagulant dose. In addition, the results of the analysis of variance obtained showed that both the full and the reduced quadratic models of the process developed were significant with p-values less than 0.0001, even though the full quadratic model contained some insignificant factors and had low prediction capability. The results, thereafter, obtained from the simulation of the modified quadratic model of the process revealed that the developed model was very good and capable of predicting the behaviour of the process well because its R-Squared, Adj R-Squared and Pred R-Squared were estimated to be 0.9281, 0.8861 and 0.6693, respectively.

## NOMENCLATURE

A	pH
Adj	Adjusted
B	Coagulant dose (g/l)
C	Coagulation time (min)
Pred	Predicted
Y	Residual turbidity (NTU)

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