International Journal of Innovative Technology and Exploring Engineering (IJITEE)
ISSN: 2278-3075 (Online), Volume-1 Issue-5, October 2012

# Modeling of Sonophotocatalytic Degradation of Formic Acid in Pharmaceutical Wastewater Treatment

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***Abstract*: Formic acid is one of the major pollutants present in pharmaceutical wastewater. The sonophotocatalytic degradation of formic acid present in pharmaceutical wastewater is analyzed in this review and mathematical model is produced for the same. The mathematical model is generated by regression analysis using Artificial Neural Network (ANN) and Support Vector Machine (SVM) to analyze & predict degradation of formic acid. This modeling and prediction helps to reduce experimental work to analyze the degradation behavior of formic acid. Modeling can also tackle insufficient data. Both ANN & SVM produced good models with negligible MSE. SVM model gave more consistent & reliable prediction compared to ANN model.***

***Index Terms*: Artificial Neural Network, Formic Acid, Sonophotocatalysis, Support Vector Machine.**

## I. INTRODUCTION

### *A. Advanced Oxidation Process (AOP)*

Because of rapid growth in industrialization, the amount of wastewater generated in industries has increased all over the globe, and the pharmaceutical industry is no exception to this. The treatment of the pollutants present in wastewater can be divided into 3 parts [1]: *physical* or primary methods like unit operations, *biological* or secondary methods like anaerobic digestion, trickling filters, etc., and *chemical* or tertiary methods like thermal oxidation (combustion), chemical oxidation, ion exchange, etc. formic acid and other lower acids are formed in the degradation or pre-oxidation step of the heavier acids i.e. longer chain aliphatic acids. Sometimes established methods of wastewater treatment cannot properly oxidize these low molecular weight pollutants [8]. Many advanced oxidation processes (AOPs) have emerged recently to remove various harmful chemicals which cannot be effectively removed from industrial wastewater by conventional treatment processes. For e.g. AOP by $O_3$/UV irradiation, Fenton's reaction, UV oxidation with $TiO_2$, US cavitation, high energy electron beam irradiation etc [4]. All AOPs have a basic chemical feature (primarily but not exclusively): the generation of highly reactive ·OH-radicals and subsequent oxidation of the pollutants by this ·OH-radicals to achieve complete mineralization of pollutants [4]. ·OH-radical is the second highest powerful oxidant after fluorine. AOPs involving ultraviolet & ultrasound irradiation can be divided as follows [3]:

***Photolysis:*** Here ultraviolet (UV) irradiation is used without any catalyst to degrade the pollutants.

*Photocatalysis:* Here UV irradiation is used in the presence of a photocatalyst (e.g. $TiO_2$, ZnO). A semiconductor material is excited by photons to produce conduction band electrons and valence band holes which induce reduction or oxidation respectively. The most effective photocatalyst for AOP is $TiO_2$ [5].

***Sonolysis:*** Breaking of chemical bonds or formation of radicals using ultrasound (US) irradiation without catalyst is called sonolysis. US irradiation produces cavitation bubbles in liquid which grow and implode in extremely small interval of time. This generates heat and pressure resulting in highly reactive radical species [3].

***Sonocatalysis:*** It is the use of US irradiation in the presence of a photocatalyst.

***Sonophotolysis:*** It is the use of US and UV irradiation simultaneously without any catalyst.

***Sonophotocatalysis:*** The use of US and UV irradiation in the presence of a photocatalyst is called sonophotocatalysis. The basic reaction mechanism here is the generation of free radicals and subsequent attack by these on the pollutant organic species. Different parameters which effect the sonophotocatalytic degradation rate are: catalyst concentration, gases dissolved in the system, catalyst pre-treatment, presence of $H_2O_2$ in the system, initial concentration of the pollutant, pH of the system, etc [2], [6], [7].

### *B. Modeling Basics*

Here we plan to use data mining process for modeling & predicting the sonophotocatalytic degradation of formic acid. Data mining refers to information extraction from large data sets and transforming it into an understandable structure by utilizing methods like artificial intelligence, machine learning, etc.

*1. Learning:* For performing data mining, we need models that 'learn' from the data. The process of adjusting parameters of the model by suitable algorithms to get desired output from the network is called learning or training, for e.g. adjusting weights in a neural network model. Learning situation can be categorized as:

*Supervised learning:* It is the task of inferring a function from available training (labeled) data. The inferred function is then expected to give correct output for the test data, for e.g. classification, regression.

**Revised Manuscript Received on 30 October 2012**
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*Unsupervised learning:* It is the task of finding patterns or structure from unlabeled data, for e.g. clustering.

*Semi-supervised learning:* It is the task of finding patterns or structure from unlabeled data with small amounts of labeled data, for e.g. finding 3-D structure of proteins.

*Reinforcement learning:* This is an intermediate of supervised & unsupervised learning. The system does some mathematical action on the environment and gets feedback. Then it evaluates the action as good or bad based on the feedback and accordingly adjusts the parameters.

*2. Regression*: Regression means to find a functional relationship between input vector and output vector. Regression analyses are done here using Artificial Neural Network (ANN) and Support Vector Machine (SVM) methods.

*3. Artificial Neural Network (ANN):* ANN is a mathematical model originally inspired by biological neural networks. Natural neuron receives signals through synapses, gets activated and emits signal through the axon if the signal surpasses a threshold value. Similarly, artificial neurons consist of inputs (like synapses), which are multiplied by weights (strength of the respective signals), and then aggregated by a mathematical function to determine the activation of the neuron.

*Feed-forward ANN:* Here, the information moves forward only in one direction: from the input nodes, through the hidden nodes and to the output nodes. The equations that rule a multilayer feed-forward ANN model are:

$$a_k = \sum w_{ki} \cdot x_i + w_{0k} \tag{1}$$

$$z_k = \sigma(a_k) \tag{2}$$

where $w_{0k}$ is bias.

The feed-forward neural network input and output neurons can be related by sigmoidal or linear type functions, as given by (3) and (4) respectively. Sigmoidal type transfer functions are necessary to introduce non-linearity in the network.

$$\sigma(s) = \frac{1}{1+e^{-s}} \tag{3}$$

$$\sigma(s) = s \tag{4}$$

*The back-propagation algorithm:* This name indicates how the weights are adjusted. It is used in many neural network models implemented in real time applications of chemical engineering processes [12]. It is a supervised learning model where the goal is to reduce training error until the network learns the training data. The ANN model begins with random weights, and then the model tries to adjust them to make the error locally minimum. The artificial neurons send their inputs 'forward', and then the errors are propagated backwards. The network error is indicated by:

$$E = \frac{1}{2}\sum_{j=1}^{q}(y_j - y_j)^2 \tag{5}$$

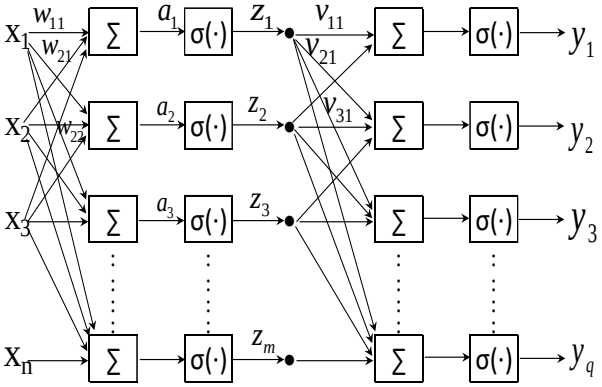


Fig.1: A multilayer feed forward artificial neural network where x & y refers to input & output respectively, w is the weight of input, a & z are intermediary outputs, $\sigma(\square)$ is the activation function.

where $y_j$ & $y_j$ are the predicted & actual (or expected) output, respectively.

How the weight changes is given by:

$$\Delta v_{jl} = -\eta \frac{\partial E}{\partial v_{jl}} \tag{6}$$

where $\eta$ (>0) is the network learning rate that controls the degree in which the gradient affects the weight changes.

It can be shown that for linear transfer function,

$$\Delta v_{jl} = \eta e_j z_l \tag{7}$$

and, for sigmoidal type transfer function,

$$\Delta v_{jl} = \eta e_j y_j (1-y_j) z_l \tag{8}$$

where $e_j = y_j - y_j$ i.e. $e_j$ is the difference between predicted & actual or expected output.

The back-propagation algorithm changes the weights as:

$$v_{jl}(n+1) = v_{jl}(n) + \Delta v_{jl} \tag{9}$$

Thus, for linear transfer function, the change in weights is expressed by:

$$v_{jl}(n+1) = v_{jl}(n) + \eta e_j z_l \tag{10}$$

and for sigmoidal type transfer function, the change in weights is expressed by:

$$v_{jl}(n+1) = v_{jl}(n) + \eta e_j y_j (1-y_j) z_l \tag{11}$$

***4. Support Vector Machine (SVM):*** SVM refers to a set of related supervised learning methods developed by Vapnik to solve classification problems. Later, it has been extended to solve regression problems too. If a training data set $\{(x_i, y_i) | 1 \le i \le l\}$ is given with input vectors $x_i$ and target concept $y_i$, with $l$ as the number of samples; then the objective of SVM regression is to find a function $f(x)$ to describe the relationship between the data set points, and later on this function is used to find output for new input data point.

$$y = f(x) = \langle \mathrm{w} \,.\, x \rangle + b \tag{12}$$

where, w is weight vector, $b$ is the bias, and <w.x> is the dot product between w and $x$.


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The regression algorithm aims to find out a flat (smooth) function to fit data. But due to experimental error (or for other reasons), practically any estimated function will have some deviation from the true function which would exactly fit the data. This deviation is known as "loss function". This can be linear, quadratic, exponential etc [16]. To avoid fitting the error values in mathematical model, Vapnik proposed "ε-insensitive loss function" to find a function *f(x)* with a maximum of ε deviation from the actually obtained targets for all the training data, and at the same time is as flat as possible. The loss function is mathematically defined as:

$$L^{\varepsilon}(y, x, f(x)) := \begin{cases} 0, & \text{if } |y - f(x)| \le \varepsilon \\ |y - f(x)| - \varepsilon, & \text{otherwise} \end{cases} \quad (13)$$

Here, deviations less than $\varepsilon$ are ignored but not otherwise.

In (12), flatness means to get a small w. For this purpose, $\|w\|^2$ *or* $\langle w.w \rangle$ can be minimized.

We can write this problem as:

$$\text{minimize} \quad \frac{1}{2}\|w\|^2$$
$$\text{subject to} \begin{cases} y_i - \langle w . x_i \rangle - b & \le \quad \varepsilon \\ \langle w . x_i \rangle + b - y_i & \le \quad \varepsilon \end{cases} \quad (14)$$

The inherent assumption here is that the problem is feasible i.e. there always exists a function which can describe all pairs ($x_i$, $y_i$) with $\varepsilon$ precision. In reality, this may not be the case always. To measure the deviation of each point outside the ε-insensitive band, Vapnik (1995) introduced two nonnegative slack variables [14] $\xi$ and $\xi_i^*$ for each data point as follows:

$$\text{minimize} \quad \frac{1}{2}\|w\|^2 + C\sum_{i=1}^{l}(\xi_i + \xi_i^*)$$
$$\text{subject to} \begin{cases} y_i - \langle w . x_i \rangle - b & \le \quad \varepsilon + \xi_i \\ \langle w . x_i \rangle + b - y_i & \le \quad \varepsilon + \xi_i^* \\ \xi_i, \xi_i^* & \ge \quad 0 \end{cases} \quad (15)$$

$C$ is the cost of errors to determine the tradeoff between the model complexity and the training errors. $\xi_i$ denotes the predicted value to be above the true value by more than $\varepsilon$ and $\xi_i^*$ to be below the true value by more than $\varepsilon$.

Equation (15) is easier to solve in its dual formulation. This also provides the key for extending SVM to nonlinear functions [14]. Lagrange multipliers are used for this dual formulation as follows:

$$L := \frac{1}{2}\|w\|^2 + C\sum_{i=1}^{l}(\xi_i + \xi_i^*) - \sum_{i=1}^{l}(\eta_i\xi_i + \eta_i^*\xi_i^*) - \sum_{i=1}^{l}\alpha_i(\varepsilon + \xi_i - y_i + \langle w . x_i \rangle + b) - \sum_{i=1}^{l}\alpha_i^*(\varepsilon + \xi_i^* - y_i - \langle w . x_i \rangle - b) \quad (16)$$

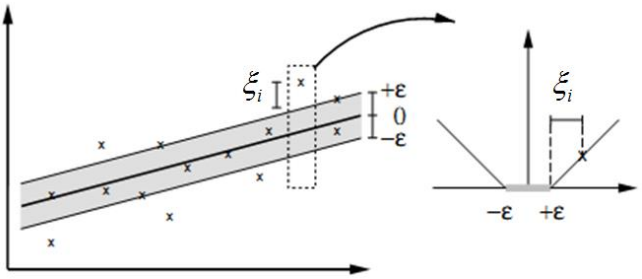


Fig.2: Soft margin loss setting for linear SVM [14]

where $L$ is the Lagrangian and $\alpha_i$, $\alpha_i^*$, $\eta_i$, and $\eta_i^*$ are Lagrange multipliers. The dual variables in (16) have to satisfy positivity constraints: $\alpha_i$, $\alpha_i^*$, $\eta_i$, $\eta_i^* \ge 0$

The partial derivatives of L with respect to the primal variables $(w, b, \xi_i, \xi_i^*)$ become 0 in optimum condition. This yields the dual optimization problem from (16):

$$\text{maximize} \begin{cases} -\frac{1}{2}\sum_{i,j=1}^{l}(\alpha_i - \alpha_i^*)(\alpha_j - \alpha_j^*)\langle x_i , x_j \rangle \\ -\varepsilon\sum_{i=1}^{l}(\alpha_i + \alpha_i^*) + \sum_{i=1}^{l} y_i(\alpha_i - \alpha_i^*) \end{cases} \quad (17)$$
$$\text{subject to} \quad \sum_{i=1}^{l}(\alpha_i - \alpha_i^*) = 0 \quad \text{and} \quad \alpha_i, \alpha_i^* \in [0, C]$$

$\alpha_i$ and $\alpha_i^*$ can be obtained by solving (17).

Also, at optimum condition,

$$\partial_\omega \mathrm{L} = \mathrm{w} - \sum_{i=1}^{l}(\alpha_i - \alpha_i^*)x_i = 0$$

Thus $\mathrm{w} = \sum_{i=1}^{l}(\alpha_i - \alpha_i^*)x_i$. We can use this in (12):

$$y = f(x) = \sum_{i=1}^{l}(\alpha_i - \alpha_i^*)\langle x_i . x \rangle + b \quad (18)$$

This is the Support Vector Machine regression expansion. Here 'w' is completely expressed as a linear combination of the training patterns $x_i$ and $\alpha_i$. 'w' need not be calculated explicitly for evaluating the function *f(x)*. Also, dot products of the data describe the whole support vector algorithm. So, it is good enough to know $k(x . x') = \langle \phi(x) . \phi(x') \rangle$ rather than to calculate $\phi$ explicitly. This allows us to restate the optimization problem for nonlinear cases as:

$$\text{maximize} \begin{cases} -\frac{1}{2}\sum_{i,j=1}^{l}(\alpha_i - \alpha_i^*)(\alpha_j - \alpha_j^*)k(x_i, x_j) \\ -\varepsilon\sum_{i=1}^{l}(\alpha_i + \alpha_i^*) + \sum_{i=1}^{l} y_i(\alpha_i - \alpha_i^*) \end{cases} \quad (19)$$
$$\text{subject to} \sum_{i=1}^{l}(\alpha_i - \alpha_i^*) = 0 \text{ and } \alpha_i, \alpha_i^* \in [0, C]$$

Similar to the linear case, Support Vector Machines regression expansion for nonlinear cases can be written as:


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$$f(x) = \sum_{i=1}^{l} (\alpha_i - \alpha_i^*)\, k(x_i . x) + b \qquad (20)$$

## II. EXPERIMENTAL PROCEDURE

In this effort we made mathematical models for analysis & prediction of sonophotocatalytic degradation of formic acid rather than finding it by experimental work. This makes tackling insufficient data easier and it saves a lot of time, money, & manpower which would have been spent in experimentation work. We reviewed the work done by P. R. Gogate, S. Mujumdar, and A. B. Pandit [2]. We referred to the degradation of formic acid by sonolysis, photocatalysis & sonophotocatalysis, and comparison of sequential & simultaneous sonophotocatalysis. The regression models were made by with Artificial Neural Networks (ANN) and Support Vector Machines (SVMs). Few studies [9], [10], [11] have supported the use of data mining techniques in making mathematical models for chemical engineering fields.

Regressive models for formic acid degradation by sonolysis, photocatalysis and sonophotocatalysis were built by taking time of irradiation, sonication input, and photocatalysis input as the input variables and % degradation of formic acid as the output from the model. To study & compare the effect of sequential and simultaneous sonophotocatalysis, we took the time of irradiation, sequential input, and simultaneous input of sonophotocatalysis as the input variables and % degradation of formic acid as the output from the model. These qualitative/categorical predictor variables (sonication input, photocatalysis input, sequential & simultaneous input of sonophotocatalysis) cannot be entered directly into a regression model and be meaningfully interpreted. So, we change them to dichotomous variables. The process of creating dichotomous variables from categorical variables is called dummy coding [17]. We assign value to each of them as 1 or 0, depending on whether they are used in the process or not. Thus it becomes useful data for ANN and SVM. For ANN, each input variable is taken as one node of input layer. Thus, we get as many input nodes as there are input variables. SVM takes input vector of the data as input and % degradation as output variable. Before using the data, we perform zero-one normalization for faster and easier training. If x is the given value of the data, then zero-one Normalization is given by:

$$x_{normalized} = \frac{(x - x_{min})}{(x_{max} - x_{min})} \qquad (21)$$

We used MATLAB Neural Network Toolbox for making feed-forward ANN models. The Levenberg-Marquardt backpropagation algorithm was used for updating weights. The function for error performance was Mean-Squared Error (MSE).

$$MSE = \frac{1}{n}\sum_{i=1}^{n}(y_{i,\,pred} - y_{i,exp})^2 \qquad (22)$$

where $y_{i,pred}$ is prediction value, $y_{i,exp}$ is experimental value, n & i refers to the number of data points and index of data, respectively.

90% of the training data was used for training the model and 10% was for validating the model. Our ANN model contains only one hidden layer. The 'number of neurons in hidden layer' (HN) was varied from 2 to 15 for find out the optimum model for given data. For each ANN structure, we performed k-fold validation and noted the minimum MSE. This way, the best ANN structure was found.

For SVM model, we used LIBSVM version 3.12 software for making the model. Here, the varying parameters were cost function (C), RBF kernel ($\gamma$), and epsilon sensitivity ($\varepsilon$). We find out the best values for C, $\gamma$, and $\varepsilon$ i.e. values that gave best MSE during cross-validation. C was varied from $2^{-1}$ to $2^{6}$; $\gamma$ was varied from $2^{-6}$ to $2^{0}$; and $\varepsilon$ was varied from $2^{-8}$ to $2^{-1}$.

## III. RESULTS AND DISCUSSION

The models confirm that sonophotocatalysis gives better results compared to the individual techniques, i.e. US & UV irradiation. The models also predict the % degradation of formic acid beyond the given data range.

### *A. ANN results to find best parameters*

We observe in Table 1 that the MSE value comes consistent from HN = 4. So, we take 4 hidden neuron layer, and the corresponding MSE = 2.4389 x $10^{-6}$.

We observe in Table 2 that the MSE value comes consistent at about HN = 5, so we take 5 hidden neuron layer, and the corresponding MSE = 1.0338 x $10^{-5}$.

Higher number of hidden neuron layers was avoided because there was no significant improvement in the result. Rather, it increases complexity of the ANN program.

Table 1: ANN results for formic acid degradation by sonolysis, photocatalysis and sonophotocatalysis

| No. of HN | MSE x $10^6$ | No. of HN | MSE x $10^6$ |
|---|---|---|---|
| 2 | 14.359 | 9 | 2.1032 |
| 3 | 2.6452 | 10 | 2.0873 |
| 4 | 2.4389 | 11 | 2.2679 |
| 5 | 2.3979 | 12 | 2.3669 |
| 6 | 3.0145 | 13 | 2.1921 |
| 7 | 2.1763 | 14 | 1.9856 |
| 8 | 2.2225 | 15 | 2.0542 |

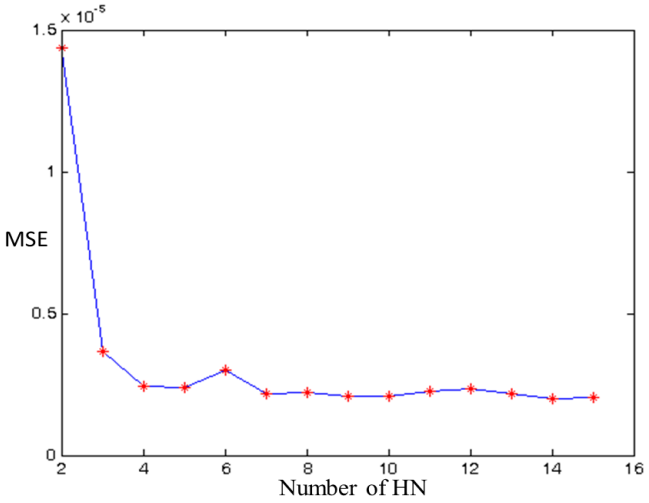


Fig.3: MSE for different number of neurons in hidden layer for formic acid degradation by sonolysis, photocatalysis and sonophotocatalysis


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Table 2: ANN results to find best parameters for formic acid degradation by sequential and simultaneous sonophotocatalysis

| No. of HN | MSE x $10^6$ | No. of HN | MSE x $10^6$ |
|---|---|---|---|
| 2 | 14.540 | 9 | 9.5386 |
| 3 | 15.227 | 10 | 9.3722 |
| 4 | 12.068 | 11 | 10.887 |
| 5 | 10.338 | 12 | 14.315 |
| 6 | 10.479 | 13 | 10.005 |
| 7 | 10.402 | 14 | 8.1272 |
| 8 | 11.93 | 15 | 9.4226 |

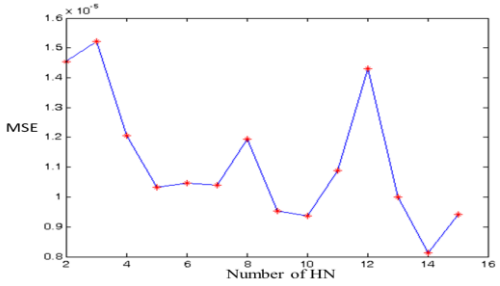


Fig.4: MSE for different number of neurons in hidden layer for formic acid degradation by sequential and simultaneous sonophotocatalysis

### *B. SVM results to find best parameters*

Table 3: Formic acid degradation by sonolysis, photocatalysis and sonophotocatalysis

| Best parameter value | | | R | MSE x $10^6$ |
|---|---|---|---|---|
| C | $\gamma$ | $\varepsilon$ | | |
| 64 | 1 | 0.00390625 | 0.999949 | 5.88052 |

Table 4: Formic acid degradation by sequential and simultaneous sonophotocatalysis

| Best parameter value | | | R | MSE x $10^6$ |
|---|---|---|---|---|
| C | $\gamma$ | $\varepsilon$ | | |
| 64 | 1 | 0.00390625 | 0.999989 | 7.7438 |

### *C. Test Results*

Table 5: MSE values for formic acid degradation by sonolysis, photocatalysis and sonophotocatalysis

| ANN | | SVM |
|---|---|---|
| HN | MSE x $10^6$ | MSE x $10^6$ |
| 4 | 2.4389 | 5.88052 |

Table 6: MSE values for formic acid degradation by sequential and simultaneous sonophotocatalysis

| ANN | | SVM |
|---|---|---|
| HN | MSE x $10^6$ | MSE x $10^6$ |
| 5 | 10.338 | 7.7438 |

## IV. CONCLUSION

Both ANN & SVM produced similar mean-squared error. We also could predict the % degradation of formic acid beyond the time specified in the data which appeared to give realistic values. Sometimes the ANN model gave relatively inaccurate prediction for degradation behavior of formic acid beyond the given data range while the SVM model gave good result consistently. So, we expect the SVM method to gain prominence in modeling & predicting similar processes.

## V. ACKNOWLEDGMENT

The authors thank Mr. Rahul Sevakula, PhD scholar, IIT Kanpur for his valuable insight which helped us in modeling.

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