Artificial neural network (ANN) approach for modeling of methyl orange adsorption by Syzygium cumini seed coat

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Abstract
In current study remediation of methyl orange was studied from aqueous media using Syzygium cumini seed coat as an active material. Batch adsorption method was utilized for the purpose and parameters optimization, including pH (3, 7 & 12), initial concentration (40, 80 & 120 ppm) and contact time (40, 80 & 120 minutes). After parameters optimization these variables were taken as input layer of Artificial Neural Network (ANN) to calculate predictive model for the removal of dye. For this purpose STATISTICA 10 software was used and architecture used for model generation was 3-8-1, which includes 3 input layers, 8 hidden and 1 output layers. Experimental results shows that Syzygium cumini seed coat is an excellent adsorber for methyl orange due to different functional groups (-OH, C=O and C-O) present on its surface as analyzed by FTIR and rough surface shown in SEM image. Predictive modeling was performed by ANN to determine adsorption capacity of the studied adsorbent for dyes. Validation of generated model was done by measuring R-square value (0.96). Results indicates Syzygium cumini can be successfully utilized for remediation of methyl orange from aqueous media and predictive modeling by ANN can predict future adsorption on this adsorbent.

Keywords: Adsorption; Artificial Neural Network (ANN); Azo dyes; Predictive modeling; Syzygium cumini

Introduction
One of the major problems faced by present world is water pollution. The discharge of industrial waste into water bodies is very harmful as it contains toxic chemicals and dyes that impart unpleasant smell and color to water [1]. Human survival is impossible without water. Out of this 3% is fresh water and just 0.06% is easily accessible to humans while the remaining consists of frozen ice caps, underground water & marshes [2]. Adsorption had been found a useful approach for elimination of dyes from industrial waste water. For this purpose wide variety of adsorbents is used that possess various characteristics depending on their chemical nature and synthesis techniques [3]. The major source of dye pollution of water bodies is the discharge of untreated textiles and waste water of leather industries that contains
various dyes. Dyes have been used by mankind from many centuries like Indigo dye was used 4000 years ago by Egyptians to envelop mummies [4]. Textile industries uses several types of dyes and the waste water produced during bleaching, washing, dyeing and finishing operations is thrown into water bodies that act as sweet poison for water bodies [5]. Dyes impart severe harms towards ecological system as they possess carcinogenic properties that makes water non suitable for the aquatic life. Actually it was the color of water bodies that pay head on the removal of dyes [6]. Various types of dyes and pigments are used by textile industries to make their product colorful and gorgeous. About 7x10^5 tons of dye material is produced per annum by the use of approximately 100,000 commercially available dyes [7]. These industries use different types of dyes such as azoic dyes, disperse dyes, solvent dyes, direct dyes, reactive dyes, acidic dyes and basic dyes. The azo dyes rank as major industrial effluents and its removal is a necessity of today [8]. There are several methods like chemical, biological and physical methods are used for the removal of dyes from industrial effluents. Even though world environmental organizations compel textile industries to remove coloring substances from waste matter before its disposal. The just removal of color from sewage does not mean that dyes are totally removed from it because the treatment process may just break down the bond of the chromophore of the dye and still the scrap of molecule remains integral so it will shift the absorption of light by the subjected molecule from visible region to Ultra Violet or Infrared region. Adsorption had been used as a physical and chemical process for the last several years and now it is evolved as a major technique for the industrial separation [9]. Food and agricultural industrial waste like rice husk, bagasse, wheat straw, orange peel, leaves, stems and coconut shells can be easily converted into adsorbents by physical and chemical means [10]. Adsorption refers to adhesion of atoms, ions or molecules to some solid surface. As a result a thin layer of adsorbate (substance that is adhered) is formed on adsorbent (the solid surface). It may be physical in which adsorption is due to Van der Waals forces between the adsorbent and adsorbate [11]. Adsorption may be chemical that is due to some chemical bond between adsorbent and the adsorbate [12]. Because of its efficiency and effectiveness adsorption is the modern day subject for removal of dyes [13]. The intention of present work is the found a cheaper alternate for the removal of methyl orange from aqueous media using various parameters.

**Materials & Methods**

In current study we utilized a commonly available agrowaste in Pakistan “Syzygium cumini” seed coat commonly called Malabar plum. Collection & pretreatment of adsorbent The fresh seed of Malabar plum were collected from different areas of Sargodha city, Pakistan. After drying the seed coat was removed from seed and washed with hot water in order to remove any sort of entrapped pigment or dye [14]. The washed sample was then dried again in an oven at 50°C over night to remove moisture. The dried seed coat was then crushed into fine powder. The conversion into fine powder is done in order to increase surface area of the adsorbent [15]. Adsorbent was then stored in air tight jar for further use.

**Characterization of sorbent**

In order to find out the efficiency of sorption process and chemical and physical parameters it is crucial to characterize the sorbent. For this purpose Fourier transform infrared spectroscopy (FTIR) and Scanning
Electron Microscopy (SEM) of the sorbent is carried out.

**Fourier transform infrared spectroscopy (FTIR)**

Fourier transform infrared spectroscopy (FTIR) is a widely used technique to determine the functional groups present in a sample. In this technique a polychromatic light is subjected on a sample and a graph is obtained. From the peaks of graph the functional groups present in a sample can be identified [16]. These functional groups provide information about active functional groups present on the surface of adsorbent for attachment of sorbate. FTIR analysis was performed in Sargodha University on Fourier Transform Infrared Spectrometer (Model Shimadzu AIM-8800).

**Scanning electron microscopy (SEM)**

Scanning Electron Microscopy (SEM) is another important technique to study surface morphology of adsorbent. It is useful to determine the presence of pores and cavities in the surface of sorbent [17]. We checked surface of our agrowaste by using this technique. Scanning Electron Microscope, JEOL model 2300 was utilized for the purpose.

**Instruments and chemicals**

Adsorption of methyl orange was determined by using UV-Vis spectrophotometer, Model Holdwell UV-8000. All the chemicals used fulfill the analytical grade. Test solutions of methyl orange were prepared by using dilution method from stock solution of 500 mg/l in distilled water.

**Adsorption experiment**

For the adsorption experiment, the amount of methyl orange adsorbed by adsorbent is investigated. Batch experimentation was done to evaluate the effect of three parameters like pH, initial concentration of dye and contact time for 100 ml of aqueous dye solution.

The solution is then filtered and the concentration of dye is determined by measuring $\lambda_{\text{max}}$ using UV-Vis spectrophotometer. The adsorbent dose and shaking speed of 1 g and 150 rpm is maintained during the batch experimentation. In order to calculate the amount of adsorbed dye following formula is used:

$$q_e = \frac{(C_0 - C_e)V}{W}$$

$q_e$, $C_0$, $C_e$, $V$ & $W$ is the amount of dye being adsorbed, initial dye concentration, volume of dye solution, weight of adsorbent [18].

**Predictive modeling and optimization**

Here we had used two types of predictive models i.e. artificial neural network (ANN) & full factorial design (FDD) for prediction of efficiency of sorbent.

**Full factorial design**

Full factorial design is useful for those experiments that take various factors into account and it is essential to study the interaction of these factors towards the net response. The main advantage of full factorial design is that it requires only small numbers of experiments for various factors thus saving the time and materials [19]. This design helps us to predict which factor shows more impact and controls the deviation of one factor over another. Here we used $2^3$ full factorial design. Three independent factors i.e. contact time; initial dye concentration and pH were varied between low, medium and high levels given in the (Table 1).

**Table 1. Factorial design applied on work**

| Factors          | Low | Med | High |
|------------------|-----|-----|------|
| $X_1$ pH         | 3   | 7   | 12   |
| $X_2$ Initial dye conc.(ppm) | 40  | 80  | 120  |
| $X_3$ Contact time(Mins)    | 40  | 80  | 120  |
Artificial neural network (ANN)
ANN is a mathematical tool to report for the efficiency of adsorption process. This model is based on human nervous system having several neurons. In this model several neurons are arranged in form of layers. These layers involve input layers, several hidden layers and one output layer. Signal is transferred from input layers to output layer through hidden layers with the help of neurons [20]. The variables used as input in the adsorption of Methyl orange are initial dye concentration (40, 80 & 120 ppm), contact time (40, 80 & 120 minutes) and PH (3, 7 & 12). Approximately 100 data points are used to nourish the structure of ANN. There are several hidden layers that perform calculations to produce net input that is then applied with activation function to produce the actual output [21]. The structure of ANN is shown in the (Fig. 1).

Figure 1. Architecture of ANN

Results and Discussion
Characterization of sorbent
For estimation of surface morphology and determination of functional groups present in the sorbent Scanning Electron Microscopy (SEM) and Fourier transform infrared spectroscopy (FTIR) is used.
Scanning electron microscopy (SEM)
For the estimation of surface morphology Scanning Electron Microscope, JEOL model 2300 was utilized for the purpose. In (Fig. 2) it shows the roughness of sorbent surface necessary for the attachment of sorbate with sorbent. From figure it is clear that the tiny pores and cavities available at the surface of sorbent are responsible for multilayer and thick adsorption of sorbate.

Fourier transform infrared spectroscopy (FTIR)
Fourier Transform Infrared Spectrometer (Model Shimadzu AIM-8800) was used for the identification of the functional groups present in Syzygium cumini seed coat. The efficiency of adsorption of sorbate by sorbent is defined by the nature of functional groups present in the sorbent [22]. In (Fig. 3) it is represented the major functional groups identified in FTIR spectra of Syzygium cumini seed coat. A broad band ranging between 500-4000 cm\(^{-1}\) may be due to stretching and bending vibrations of C=O, O-H and C-H groups. Stretching band of C-H appears between ranges of 3000-3300 cm\(^{-1}\). Peaks appearing between 2500-3500 cm\(^{-1}\) are
due to O-H group while peak appearing at 1750 cm$^{-1}$ is due to C=O group. As C=O, O-H and C-H functional groups are polar in nature so they can form electrostatic interactions with polar groups present in methyl orange and adsorption occurs. The FTIR of Malabar plum seed coat and structure of methyl orange is shown in (Fig. 3 & 4).

Figure 2. SEM image of raw Syzygium cumini at four different resolutions

Figure 3. FTIR Spectra of Syzygium cumini
Predictive modeling by full factorial design

To check the effect of three parameters on the adsorption of methyl orange onto *Syzygium cumini* [23], full factorial design is adopted. The predicted response is denoted by the equation given below;

\[ Y = b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_1X_2 - b_5X_1X_3 - b_6X_2X_3 + b_7X_1X_2X_3 \]

Here \( Y \) represents predicted response; \( b_0 \) represents intercepts while \( X_1, X_2 \) and \( X_3 \) represents pH, initial dye concentration and contact time respectively while their products in different combinations represent the interaction of parameters with one another [23]. Predicted response was calculated by using linear model proposed in equation 1. The comparison of predicted response and experimental response along with % variance was presented in the (Table 2). The closeness of calculated response with experimental response confirmed the validity of model for current study. These results were also examined by ANOVA. Coefficients of all the possible combinations of variables, their standard error, t-stat & p-value were calculated. The p-value is useful to estimate the effect of individual parameter and its interaction with other parameters. Smaller is the p-value more significant is the variable for calculation of response. In this context \( X_2 \& X_3 \) were found more significant towards the adsorption of sorbate. The estimated effects and coefficients for R and statistical parameters including coefficients, standard error, t-stat and p-value were shown in (Table 3 & 4) respectively.

Artificial neural network (ANN) for predictive modeling

Artificial Neural Network (ANN) is a mathematical tool to evaluate the efficiency of adsorption process. The variables used as input in the adsorption of Methyl orange are pH (3, 7 & 12), initial dye concentration (40, 80 & 120 ppm) and contact time (40, 80 & 120 minutes). For this presented work a three input layer ANN i.e. \( X_1 \) (pH), \( X_2 \) (initial dye concentration) and \( X_3 \) (Contact time), 3-10 hidden layers and one output layer i.e. % sorption is designed. For *Syzygium cumini* best response is obtained by 3 input layers, 5 hidden layers and one output layer. ANN is a better model providing favorable solution and understanding to explain the actual performance of adsorption process [24]. Design matrix and result of ANN model for adsorption of Methyl orange onto *Syzygium cumini* (Malabar plum) seed coat is given below in (Table 5).

From the above table it is clear that the best response was obtained by 3 input layers, 5 hidden layers and one output layer. Although best response is obtained by highest number of hidden layers but it is not necessary that highest number of hidden layers always give best response.
In (Table 6) it is represented comparison of experimental response; FFD calculated response & ANN predicted response along with % variance.

Table 2. Comparison of predicted response and experimental response along with % variance

| Runs | X1 pH | X2 Initial conc. (ppm) | X3 Time (min) | Experimental Response | Calculated Response | % Variance |
|------|-------|------------------------|---------------|-----------------------|---------------------|------------|
| 1    | 3     | 40                     | 40            | 23.6                  | 21.52862096        | 2.07137904 |
| 2    | 3     | 80                     | 40            | 42.8625               | 39.77206628        | 3.09043372 |
| 3    | 3     | 120                    | 40            | 52.18333333          | 58.01551159        | -5.83217826 |
| 4    | 7     | 40                     | 40            | 2.5                   | 25.70439331        | -23.2043933 |
| 5    | 7     | 80                     | 40            | 6.75                  | 41.49540359        | -34.7454036 |
| 6    | 7     | 120                    | 40            | 48.41666667          | 57.28641387        | -8.86974721 |
| 7    | 12    | 40                     | 40            | 3.175                 | 30.92410875        | -27.7491088 |
| 8    | 12    | 80                     | 40            | 15.9375               | 43.64957524        | -27.7120752 |
| 9    | 12    | 120                    | 40            | 22.11666667          | 56.37504173        | -34.283751 |
| 10   | 3     | 40                     | 80            | 24.375                | 22.90226091        | 1.47273909 |
| 11   | 3     | 80                     | 80            | 43.8375               | 40.5427187         | 3.2947813  |
| 12   | 3     | 120                    | 80            | 52.23333333          | 58.18317649        | -5.94984315 |
| 13   | 7     | 40                     | 80            | 5.45                  | 27.30079823        | -21.8507982 |
| 14   | 7     | 80                     | 80            | 7.35                  | 42.56921716        | -35.2192172 |
| 15   | 7     | 120                    | 80            | 49.19166667          | 57.83763609        | -8.64596942 |
| 16   | 12    | 40                     | 80            | 3.325                 | 32.79896987        | -29.4739699 |
| 17   | 12    | 80                     | 80            | 16.4125               | 45.10234023        | -28.6898402 |
| 18   | 12    | 120                    | 80            | 22.375                | 57.40571059        | -35.0370106 |
| 19   | 3     | 40                     | 120           | 24.475                | 24.27590086        | 0.19909914 |
| 20   | 3     | 80                     | 120           | 43.9375               | 41.31337112        | 2.62412888 |
| 21   | 3     | 120                    | 120           | 52.4                  | 58.35084139        | -5.95084139 |
| 22   | 7     | 40                     | 120           | 8.7                   | 28.89720314        | -20.1972031 |
| 23   | 7     | 80                     | 120           | 7.4625                | 43.64303072        | -36.1805307 |
| 24   | 7     | 120                    | 120           | 49.4                  | 58.3888583         | -8.9888583  |
| 25   | 12    | 40                     | 120           | 3.825                 | 34.673831          | -30.848831 |
| 26   | 12    | 80                     | 120           | 17.6125               | 46.55510522        | -28.9426052 |
| 27   | 12    | 120                    | 120           | 22.48333333          | 58.43637945        | -35.9530461 |

Table 3. Estimated effects and coefficients for R

| Terms      | Coefficients | Standard Error | t Stat   | P-value |
|------------|--------------|----------------|----------|---------|
| Intercept  | 0.87933271   | 21.12591142    | 0.041623421 | 0.967233196 |
| X1         | 0.047548118  | 0.2509332      | 0.85172215 | 0.389458163 |
| X2         | -1.58562282  | 3.241069162    | -0.48922831 | 0.630280077 |
| X3         | 0.486772512  | 0.2509332      | 1.939848982 | 0.06738939 |
| X1X2       | -0.0009194   | 0.038497362    | -0.023882177 | 0.981195632 |
| X1X3       | -0.00038831  | 0.00298058     | -0.13028143 | 0.89771323 |
| X2X3       | -0.01579802  | 0.038497362    | -0.41036618 | 0.686127912 |
| X1X2X3     | 1.25619E-05  | 0.000457271    | 0.027471462 | 0.978370197 |
Table 4. Statistical parameters for $3^3$ designs

| Regression Statistics   |       |
|-------------------------|-------|
| Multiple R              | 0.900249788 |
| R Square                | 0.810449681 |
| Adjusted R Square       | 0.740615353 |
| Standard Error          | 9.331316186 |
| Observations            | 27    |

Table 5. Design Matrix and Results of ANN Model for Adsorption of Methyl Orange onto Syzygium cumini

| Sample | % sorption= Cad/Ci*100 | % sorption= Cad/Ci*100 - Output | % sorption= Cad/Ci*100 - Output | % sorption= Cad/Ci*100 - Output | % sorption= Cad/Ci*100 - Output |
|--------|------------------------|---------------------------------|---------------------------------|---------------------------------|---------------------------------|
| Test   | 23.60                  | 22.16                           | 22.94                           | 23.81                           | 23.93                           |
| Train  | 42.86                  | 43.95                           | 42.29                           | 41.50                           | 42.89                           |
| Test   | 52.18                  | 51.57                           | 52.88                           | 50.81                           | 50.80                           |
| Train  | 2.50                   | 3.22                            | 2.95                            | 2.17                            | 5.30                            |
| Train  | 6.75                   | 7.25                            | 6.84                            | 8.26                            | 6.70                            |
| Train  | 48.41                  | 48.85                           | 48.43                           | 49.25                           | 48.45                           |
| Test   | 3.17                   | 3.80                            | 2.84                            | 3.01                            | 4.69                            |
| Train  | 15.93                  | 15.21                           | 15.42                           | 15.38                           | 16.42                           |
| Train  | 22.11                  | 21.89                           | 22.07                           | 21.15                           | 22.45                           |
| Train  | 24.37                  | 23.63                           | 24.08                           | 24.56                           | 24.20                           |
| Train  | 43.83                  | 43.60                           | 43.27                           | 43.21                           | 43.31                           |
| Train  | 52.23                  | 51.52                           | 52.76                           | 52.34                           | 51.84                           |
| Train  | 5.45                   | 5.41                            | 5.18                            | 4.59                            | 5.55                            |
| Train  | 7.35                   | 7.30                            | 7.90                            | 8.69                            | 6.90                            |
| Train  | 49.19                  | 48.56                           | 48.81                           | 48.92                           | 48.98                           |
| Train  | 3.32                   | 3.86                            | 3.65                            | 3.84                            | 4.73                            |
| Test   | 16.41                  | 16.76                           | 16.01                           | 15.98                           | 16.62                           |
| Train  | 22.37                  | 22.34                           | 22.17                           | 22.23                           | 22.64                           |
| Train  | 24.47                  | 25.14                           | 24.73                           | 24.83                           | 24.48                           |
| Train  | 43.93                  | 43.28                           | 43.75                           | 44.42                           | 43.75                           |
| Train  | 52.40                  | 51.47                           | 52.29                           | 53.77                           | 53.04                           |
| Train  | 8.70                   | 7.37                            | 7.60                            | 6.90                            | 5.82                            |
| Test   | 7.46                   | 7.65                            | 8.76                            | 8.82                            | 7.11                            |
| Train  | 49.40                  | 48.24                           | 48.67                           | 47.95                           | 49.64                           |
| Train  | 3.82                   | 3.81                            | 5.05                            | 5.05                            | 4.76                            |
| Train  | 17.61                  | 18.17                           | 17.19                           | 16.75                           | 16.75                           |
| Train  | 22.48                  | 22.95                           | 22.62                           | 22.89                           | 22.76                           |
Table 6. Comparison of Predicted Response & % Variance of RSM & ANN Models for Syzygium cumini

| X1 | X2  | X3  | Exp. Resp | FFD predicted. Resp | % Variance | ANN predicted. Resp | % Variance |
|----|-----|-----|-----------|---------------------|------------|---------------------|------------|
| 3  | 40  | 40  | 23.6      | 21.52               | 2.07       | 23.93               | 0.51       |
| 3  | 80  | 40  | 42.86     | 39.77               | 3.09       | 42.89               | 0.08       |
| 3  | 120 | 40  | 52.18     | 58.01               | -5.83      | 50.80               | 0.85       |
| 7  | 40  | 2.50 | 25.70     | -23.20              | 5.30       | -0.70               |
| 7  | 80  | 6.75 | 41.49     | -34.74              | 6.70       | -0.14               |
| 7  | 120 | 48.41 | 57.28     | -8.86               | 48.45      | -0.45               |
| 12 | 40  | 3.17 | 30.92     | -27.74              | 4.69       | 0.82                |
| 12 | 80  | 15.93 | 43.64     | -27.71              | 16.42      | 0.11                |
| 12 | 120 | 22.11 | 56.37     | -34.25              | 22.45      | -0.08               |
| 3  | 40  | 24.37 | 22.90     | 1.47                | 24.20      | 0.50                |
| 3  | 80  | 43.83 | 40.54     | 3.29                | 43.31      | 0.29                |
| 3  | 120 | 52.23 | 58.18     | -5.94               | 51.84      | 0.12                |
| 7  | 40  | 5.45  | 27.30     | -21.85              | 5.55       | 0.44                |
| 7  | 80  | 7.35  | 42.56     | -35.21              | 6.90       | 0.29                |
| 7  | 120 | 49.19 | 57.83     | -8.64               | 48.98      | 0.46                |
| 12 | 40  | 3.32  | 32.79     | -29.47              | 4.73       | 0.46                |
| 12 | 80  | 16.41 | 45.10     | -28.68              | 16.62      | 0.61                |
| 12 | 120 | 22.37 | 57.40     | -35.03              | 22.64      | 0.01                |
| 3  | 40  | 24.47 | 24.27     | 0.19                | 24.48      | -0.50               |
| 3  | 80  | 43.93 | 41.31     | 2.62                | 43.75      | -0.64               |
| 3  | 120 | 52.40 | 58.35     | -5.95               | 53.04      | -0.71               |
| 7  | 40  | 8.70  | 28.89     | -20.19              | 5.82       | 1.16                |
| 7  | 80  | 7.46  | 43.64     | -36.18              | 7.11       | -0.43               |
| 7  | 120 | 49.40 | 58.38     | -8.98               | 49.64      | 0.37                |
| 12 | 40  | 3.82  | 34.67     | -30.84              | 4.76       | -0.60               |
| 12 | 80  | 17.61 | 46.55     | -28.94              | 16.75      | 0.82                |
| 12 | 120 | 22.48 | 58.43     | -35.95              | 22.76      | -0.90               |

The value of FFD predicted response and ANN predicted response is slightly different. But % variances calculated for both FFD and ANN model show that the value of % variance is low for ANN. Smaller the value of % variance means that experimental and calculated responses are close to each other. 

As % variance for ANN is small so it is better fit model then SEM for modeling of % sorption of methyl orange onto Malabar plum. It is also evident from the (Fig. 5 & 6) that the experiment response is more closer to ANN calculated response than FFD calculated response.
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**Conclusion**

Removal of *Methyl orange by means of Syzygium cumini* was successfully performed. Efficiency of the adsorbent was checked experimentally as well as predicted by ANN, and closeness of experimental and predicted results shows success of generated ANN model for current work. Surface morphology and functional groups analysis also support the results. This study is a fruitful addition in pool of adsorption study and predictive modeling.

**Authors’ contributions**

Conceived and designed the experiments: F Batool, Performed the experiments: F Abbas & T Iqbal, Analyzed the data: S Iqbal & J Akbar, Contributed materials/ analysis/ tools: S Noreen, Wrote the paper: F Batool & Faisal Iqbal.
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