# Response surface methodology for optimization of the adsorption capability of ball-milled pomegranate peel for different pollutants 

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#### Abstract

Pomegranate peel waste was milled using ball milling to get particles of size of $90 \mu \mathrm{~m}$ and used for adsorption of toxic pollutants. The response surface methodology was used for modeling of the adsorption capacity of copper ions and C.I. Reactive Yellow 145 (3RS) from wastewater using ball-milled pomegranate peel (BMPP). The predicted removal capacities obtained from the model are in a good agreement within the experimental results with correlation regression of 0.94 for dye adsorption and 0.92 for metal adsorption. The maximum removal capacity over adsorbent biomass is expected to be $209.7103 \mathrm{mg} \mathrm{g}^{-1}$ for dye and $103 \mathrm{mg} \mathrm{g}^{-1}$ for metal at optimal values of the independent variables. The analogue experimental results are $224 \mathrm{mg} \mathrm{g}^{-1}$ for dye and $106 \mathrm{mg} \mathrm{g}^{-1}$ for metal. Conclusively, the obtained results confirm the applicability of used model for predicting the adsorptive capacity of ball-milled pomegranate peel of dye and metals. As well ball-milled pomegranate peel is considering a powerful, cost effective and anionic/cationic adsorbent for decontamination of wastewater.


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## 1. Introduction

Deterioration of water quality is mostly originated from untalented utilization of natural water, and extensive urbanization and industrialization. It has a harmful impact on human health and environment. Direct/indirect discharge of wastewater containing non-degradable dyes and toxic metals into water bodies are the most serious environmental problems [1-3]. Hence, the principal crux target of environmental researchers is development of the feasible, green and economic remediation procedures for decontamination of pollutants from environment devoid of any harmful impact on human health [4-6]. Various remediation techniques are implemented for control of aquatic pollution [7]. Most of them have some drawbacks; such as high cost, low efficiency for removal of emanating pollutants and generation of risky sludge [8-10]. The immediate challenge is finding low-cost treatment methods for removal of dye and metals from wastewater. Adsorption process was applied for decontamination of these pollutants using biomass. Optimization of treatment process is the needed fundamental mission for researchers. However, minimizing the

[^0]number of experiments is required for decreasing the cost, the consumed period, and the environmental effect. The response surface method (RSM) is a commanding procedure which could accomplish to optimize the treatment process. RSM is a combination of statistical and mathematical models for creating, improving and optimizing the processes by employing a set of designed experiments. It is an illustration of the variations of the response (i.e. efficiency or removal) as a function of two independent variables at the central value of the other variables. While, the contour plot was a 2D plane graph where each line in the 2D contour plot accounts for infinite combinations of two independent variables at the constant value of surface response. The contour plot is very useful for estimation of the interaction between different input variables and identification their optimal levels [11-14]. The design of the experiments (DoE) is an essential part of the RSM for evaluation of the factors that have significant effect on the response. Moreover, DoE select the number of levels for each factor and there combinations with other factors at which the response should be evaluated [15]. Herein, pomegranate peel is milled using ball milling to enhance the adsorption process of $\mathrm{Cu}^{2+}$ ions and dye from wastewater. As well, the current work will construct models for treatment of non-degradable dyes and toxic copper ions using RSM and the central composite design.


Fig. 1. C.I. reactive yellow 145 (3RS).

Table 1
Input variables and there levels employed in the $2^{4}$ central composite design.

| Variables | Range of values and levels |  |  |  |  |
| :--- | :--- | :--- | :--- | :--- | :--- |
|  | -2 | -1 | 0 | 1 | 2 |
| $X_{1}$, time (min) | 30 | 60 | 90 | 120 | 150 |
| $X_{2}, \mathrm{pH}$ | 1 | 2 | 3 | 4 | 5 |
| $X_{3}$, dose $\left(\mathrm{g} \mathrm{L}^{-1}\right)$ | 0.2 | 0.5 | 0.8 | 1.1 | 1.4 |
| $X_{4}$, init. concentration $\left(\mathrm{mg} \mathrm{L}^{41}\right)$ | 25 | 50 | 75 | 100 | 125 |

## 2. Materials and experimental design

### 2.1. Materials used

Materials used in the study are including the following; C.I. Reactive Yellow 145 (3RS) (Fig. 1) with Molecular Formula: $\mathrm{C}_{28} \mathrm{H}_{20} \mathrm{Cl}$
$\mathrm{N}_{9} \mathrm{Na}_{4} \mathrm{O}_{16} \mathrm{~S}_{5}$ and Molecular Weight: 1026.25 were obtained from local textile factory (Cairo, Egypt). Copper (II) sulphate pentahydrate $\left(\mathrm{CuSO}_{4} \cdot 5 \mathrm{H}_{2} \mathrm{O}\right)$ from Merck (Merck, Germany) was used for preparation of stock solution of copper ions solution in deionized water.

### 2.2. Ball milling of pomegranate peel

Pomegranate peels were obtained from the waste of juice manufacturing Factory. These peels were washed to remove any impurities, and dried at $60{ }^{\circ} \mathrm{C}$. Then they were crushed with up to grain size of $3-6 \mathrm{~mm}$ and then washed again several times with deionized water until no color was observed. Then, it was dried and mechanically milled using a planetary ball mill (Retsch PM 100 CM, Germany). In a typical procedure, 10 g of starting preliminary material was loaded together with several stainless steel balls of 10 mm diameters (powder/ball $=1 / 10$ weight ratio) placed in a 500 mL stainless steel container then start grinding process at 50 rpm for 1 h . The fine powder was collected and passed through suitable sieve with pore size $90 \mu \mathrm{~m}$.

### 2.3. Characterization of ball milled-pomegranate peel

FTIR spectrophotometer (630-JASCO, JASCO, Japan) was used to determine the differences in functional groups before and after the sorption process on BMPP. Pellets were prepared with 2 mg BMPP biomass and 100 mg KBr . The spectra were recorded in the wave number range of 400 to $4000 \mathrm{~cm}^{-1}$. The specific surface area, pore size and pore volume of the samples were measured using nitrogen sorption (NOVA touch instrument, Quantachrome, USA) at 77 K . Prior to the sorptometric experiment, the samples were degassed at 423 K for 12 h . Spectrum acquisition and analysis

Table 2
Central composite design for the experiment and the response results.

| Run | $X_{1}$ | $X_{2}$ | $X_{3}$ | $X_{4}$ | Time (min) | $\mathrm{pH}(-)$ | Dose ( $\mathrm{L} \mathrm{L}^{-1}$ ) | Initial conc. ( $\mathrm{mg} \mathrm{L}^{-1}$ ) | Adsorbed dye ( $\mathrm{mg} \mathrm{g}^{-1}$ ) |  | Adsorbed metal (mg $\mathrm{g}^{-1}$ ) |  |
| :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: | :---: |
|  |  |  |  |  |  |  |  |  | Observed | Predicted | Observed | Predicted |
| 1 | -1 | -1 | -1 | -1 | 60 | 2 | 0.5 | 50 | 71.92 | 67.4493 | 5 | 6.5850 |
| 2 | -1 | -1 | -1 | 1 | 60 | 2 | 0.5 | 100 | 105.26 | 98.5008 | 9.4 | 17.2392 |
| 3 | $-1$ | $-1$ | 1 | -1 | 60 | 2 | 1.1 | 50 | 35.90 | 33.2510 | 2.73 | 6.7208 |
| 4 | -1 | -1 | 1 | 1 | 60 | 2 | 1.1 | 100 | 36.65 | 32.9844 | 1.36 | 6.3900 |
| 5 | -1 | 1 | -1 | -1 | 60 | 4 | 0.5 | 50 | 41.72 | 47.9922 | 39 | 37.9425 |
| 6 | -1 | 1 | -1 | 1 | 60 | 4 | 0.5 | 100 | 97.64 | 97.7762 | 58 | 58.6617 |
| 7 | -1 | 1 | 1 | -1 | 60 | 4 | 1.1 | 50 | 18.40 | 8.4001 | 17.73 | 17.4733 |
| 8 | -1 | 1 | 1 | 1 | 60 | 4 | 1.1 | 100 | 20.17 | 26.8659 | 23.64 | 27.2075 |
| 9 | 1 | -1 | -1 | -1 | 120 | 2 | 0.5 | 50 | 88.23 | 69.9874 | 7 | 8.2142 |
| 10 | 1 | -1 | -1 | 1 | 120 | 2 | 0.5 | 100 | 86.83 | 97.0864 | 15.8 | 21.0133 |
| 11 | 1 | -1 | 1 | -1 | 120 | 2 | 1.1 | 50 | 40.10 | 40.2205 | 2.18 | 6.4750 |
| 12 | 1 | -1 | 1 | 1 | 120 | 2 | 1.1 | 100 | 53.82 | 36.0013 | 2.45 | 8.2892 |
| 13 | 1 | 1 | -1 | -1 | 120 | 4 | 0.5 | 50 | 44.45 | 48.3717 | 38 | 37.9267 |
| 14 | 1 | 1 | -1 | 1 | 120 | 4 | 0.5 | 100 | 103.10 | 94.2032 | 60 | 60.7908 |
| 15 | 1 | 1 | 1 | -1 | 120 | 4 | 1.1 | 50 | 18.00 | 13.2110 | 18.64 | 15.5825 |
| 16 | 1 | 1 | 1 | 1 | 120 | 4 | 1.1 | 100 | 23.00 | 27.7243 | 24.09 | 27.4617 |
| 17 | $-2$ | 0 | 0 | 0 | 30 | 3 | 0.8 | 75 | 29.01 | 30.5863 | 23.75 | 17.9392 |
| 18 | 2 | 0 | 0 | 0 | 150 | 3 | 0.8 | 75 | 24.27 | 33.9827 | 23.75 | 19.8225 |
| 19 | 0 | -2 | 0 | 0 | 90 | 1 | 0.8 | 75 | 33.20 | 49.1625 | 2.5 | 0 |
| 20 | 0 | 2 | 0 | 0 | 90 | 5 | 0.8 | 75 | 26.11 | 21.4284 | 37.5 | 40.3958 |
| 21 | 0 | 0 | -2 | 0 | 90 | 3 | 0.2 | 75 | 128.46 | 131.7081 | 45.6 | 42.3825 |
| 22 | 0 | 0 | 2 | 0 | 90 | 3 | 1.4 | 75 | 22.99 | 31.0310 | 15.71 | 9.1892 |
| 23 | 0 | 0 | 0 | -2 | 90 | 3 | 0.8 | 25 | 29.00 | 38.2745 | 18.38 | 19.9292 |
| 24 | 0 | 0 | 0 | 2 | 90 | 3 | 0.8 | 125 | 81.83 | 83.8393 | 53.75 | 42.4625 |
| 25 | 0 | 0 | 0 | 0 | 90 | 3 | 0.8 | 75 | 42.80 | 41.3161 | 31.25 | 31.9817 |
| 26 | 0 | 0 | 0 | 0 | 90 | 3 | 0.8 | 75 | 37.77 | 41.3161 | 32.5 | 31.9817 |
| 27 | 0 | 0 | 0 | 0 | 90 | 3 | 0.8 | 75 | 41.50 | 41.3161 | 33.13 | 31.9817 |
| 28 | 0 | 0 | 0 | 0 | 90 | 3 | 0.8 | 75 | 40.23 | 41.3161 | 31.88 | 31.9817 |
| 29 | 0 | 0 | 0 | 0 | 90 | 3 | 0.8 | 75 | 42.80 | 41.3161 | 30.63 | 31.9817 |
| 30 | 0 | 0 | 0 | 0 | 90 | 3 | 0.8 | 75 | 42.80 | 41.3161 | 32.5 | 31.9817 |

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