Study on different parts of moringa oleifera for treating wastewater

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Abstract. In this study Moringa Oleifera (MO) leaves and pods are utilized for the treatment of Methylene Blue (MB) dye solution. The uptake of pollutants by MO leaves and pods are assessed by varying dosages from 0.01g to 0.1g with different agitation time of 5min to 30 min. From the observed results, the maximum removal (96%) occurs by adding MO leaves at 0.08g dosage at the time period of 15minutes. MO pods removes maximum of 45% at the dosage of 0.1g at 30 minutes. It is found that MO leaves are better in removing organic dye than MO pods. The Scanning Electron Microscope (SEM) and Energy Dispersive X Ray (EDX) analysis are carried out to identify the morphological character and elemental composition of the MO leaves and pods. The result shows that the leaves contain more carbon content (33%) than pods (20.3%). Similarly the amount of oxygen is least in leaves thus enhancing the removal of dye particles. The SEM result indicates that the pore development is more in leaves than pods. Finally, Isotherm model was developed for pods and leaves; it is found that MO pods follow Freundlich equation whereas MO leaves follow Langmuir equation.

1. Introduction

Water is the essential resource for all living organism to thrive, in this modern era water scarcity and water pollution are the most serious challenges being faced globally. Natural water bodies are getting polluted by the discharge of industrial and domestic wastes, many rivers lost its self-purification ability due to excessive pollution and on the other hand many of the natural water bodies disappeared due to urbanization.

India has almost 6.3 crores of Micro, Small and Medium scale enterprises (MSME). Tamilnadu has largest number of MSME and it covers almost 15% of the country, 6.89 lakhs registered MSMEs, producing over 8,000 varieties of product for a total investment of more than Rs. 32,008 crores. The biggest challenge in MSME is to treat the wastewater. The highly complicated methods of treatment are not affordable by small and medium scale industries. To overcome these issues, Common Effluent Treatment Plant (CETP) has been installed in many parts of the country to minimize the cost of treatment, land cost, and provide better collective treatment for small scale industrial facilities [1]. The major challenge in CETP is to manage shock loads such as sudden change in hydraulic loads and organic loads cause failure of many CETPs [1]. Some of the industries are not located in industrial region and it
is located at isolated region. There is a need an alternative, sustainable, very simple affordable technique for treating Industrial wastewater for isolated small scale industries. Moringa oleifera (MO) is harmless and low-priced adsorbent, it can be used for both drinking and wastewater treatment and it is used as promising and primary coagulant as well as adsorbent [2-7]. MO is a tropical plant belongs to the family Moringaceae, mostly used as a vegetable, medicines, oil purposes [8] and it is native to India and it is found abundantly throughout the tropics [9].

In previous researches, MO seed kernel is used for water treatment. It is a known fact that the availability of seed kernel is very less than drum stick pods, leaves and barks. This study aims to identify the effectiveness of other parts of the MO namely leaves and pods for treating synthetic wastewater by varying the dosages and agitation time period. Comparison is made between MO leaves and pods for the removal of methylene blue solution. Elemental composition (EDX) of pods and leaves are carried for identifying the major elements contributing the removal of dye particles. Micro structural characteristics namely SEM images of adsorbents are analysed to understand the changes in the structure before and after treatment. Finally an isotherm model is developed for MO leaves and pod.

2. Methodology
The dried moringa pods and leaves are collected from nearby garden and dried in the natural sunlight to remove the moisture content completely. The pod is peeled from dried flesh and kernels and grinded into a fine powder. The powdered pod is kept in a muffle furnace at 350°C for 2 hours to activate the pod powder. The activated powder is then taken through a series of cleaning process by using distilled water to remove unwanted dissolved waste materials. The cleaned activated pod is then kept in a hot air oven at 105°C until the moisture is completely removed (Bello S et al. 2014). The dried activated pod is then grinded in a crush along with mortar and pestle and used for treatment. The same procedure is adopted for leaves also.

2.1 Preparation of synthetic wastewater
Methylene Blue (MB), cationic dye is used in dyeing industries such textile industry for different purposes dyeing and cloth completion processes [10]. In this study methylene blue dye is prepared and it is basically organic dye and the chemical formula of methylene blue dye is C₁₆H₁₈CIN₃S. It is already used for MO treatment using kernel [11].

2.2 Experimental setup
In this work, two case studies are undertaken to assess the capacity of removal of synthetic dye using MO leaves and MO pods by changing adsorbent dosage and contact time. Different trials are made by varying the proportion of adsorbent dosage from 0.01g to 0.1g, upon obtaining the optimum dosage level the effective contact time is determined by repeating the trails at various time intervals. The different dosage quantity of activated leaf carbon from 0.01g to 0.1g is added in 100 ml of sample. All conical flasks are tightly covered with aluminium foil and kept in a mechanical shake for 30 minutes. The absorbance values are measured for all the dosage levels and the optimum dosage is identified, subsequently the time is varied from 5 to 30 minutes to identify the optimum time duration for removal of dye particles. Similarly the same procedure is adopted for pod as an adsorbent.

2.3 Scanning Electron Microscope (SEM) and Energy Dispersive X ray Analysis (EDX)
Scanning Electron Microscope (SEM) analysis is used to study the surface morphology of the adsorbent before and after the adsorption and it also gives an idea of how much adsorbate is attached on adsorbent. EDX is used to identify the elements present in Moringa oleifera. The characteristics element is identified based upon the energy value of photon. The peak indicates in the spectrum shows the concentration of the particular element.
2.4 Adsorption study
The process of adsorption is usually studied through adsorption isotherm. It is the graph between the amount of adsorbate (X) adsorbed on the surface of adsorbent (M). In this study, Freundlich and Langmuir adsorption isotherm studies are used for the adsorption process.

2.5 Freundlich isotherm
The mass of the gas adsorbed per gram of the adsorbent is plotted against pressure in the form of a curve to show the relationship. Here, at a fixed pressure, physical adsorption decreases with increase in temperature. The curves reach saturation at high pressure. Equation (1) shows Freundlich equation

\[ \log \frac{x}{m} = \log K + \frac{1}{n} \log C_e \]  

\( \frac{x}{m} \) = mass of adsorbate adsorbed per unit mass of adsorbent (mg of adsorbate/g of adsorbent)

\( C_e \) = equilibrium concentration of adsorbate in solution after adsorption (mg/l)

\( K_f \) and \( n \) are Freundlich constants

\( \frac{1}{n} \) is the slope of the equation and \( K_f \) is the intercept obtained from graph

The graph is plotted with \( \log C_e \) and \( \log \frac{x}{m} \) and the constants were found

2.6 Langmuir Adsorption Isotherm
The Langmuir adsorption isotherm describes the surface as homogeneous, assuming that there is no lateral interaction between adjacent adsorbed molecules when a single molecule occupies a single surface site. Equation (2) shows Langmuir equation

\[ \frac{C_e}{Q_e} = \frac{1}{bQ_o} + \frac{C_e}{Q_o} \]  

\( C_e \) = equilibrium concentration of adsorbate in solution after adsorption mg/L

\( Q_e = \frac{x}{m} \) = mass of adsorbate adsorbed per unit mass of adsorbent (mg/g)

\( b \) and \( \frac{1}{Q_o} \) are Langmuir constants

The graph is plotted with \( C_e \) and \( \frac{C_e}{Q_e} \) and constants were found

Where \( b \) is the intercept and \( \frac{1}{Q_o} \) is the slope obtained from the plotted graph

3. Results and Discussions
3.1 Effect of adsorbent dosage on removal of pollutants
Table 1 shows the concentration of MB of two case studies by changing adsorbent dosages. It is observed from table 1, 2 and figure 1, almost 90% removal takes place at 0.01g itself while using moringa leaves whereas pod removes almost 43% at 0.1 g dosage. It is found that ten times more adsorbent dosage of pods (0.1g) used, the removal is only half of the leaves. The maximum removal of 96% is obtained at the dosage of 0.08g/100ml in leaves whereas pods the maximum removal is 43% at 0.1g/100ml. The maximum removal in leaves may be due to more number of active sites are present in adsorbing pollutants. Initially the removal is faster in leaves because of more vacant sites. After accumulation of pollutants on vacant sites, removal becomes slower and reaches to a maximum of 96%. When comparing to leaves, the percentage removal is less while using pod as an adsorbent. The contributing factors for
decrease in performance while using pods may be due to number of factors such as improper formation of fine pores within the material, minimum surface area etc [12].

Then optimum dosage of adsorbent is fixed and the contact time is changed. Table 2 and figure 1 shows the effect contact time on removal efficiency. From table 2, it is found that pod having the maximum removal efficiency of 40% at a time period of 30 minutes whereas leaves having the removal efficiency of more than 90% at a time period of 15 minutes. So it recommended using leaves at the dosage of 0.08 g at a contact period of 15 minutes.

| Dosage (g) | Concentration (mg/l) Pods | Concentration (mg/l) leaves | Removal (%) Pods | Removal (%) leaves |
|-----------|---------------------------|-----------------------------|-----------------|-------------------|
| 0         | 0.35                      | 0.35                        | 0               | 0                 |
| 0.01      | 0.32                      | 0.040                       | 8.57            | 88.57             |
| 0.02      | 0.3                       | 0.035                       | 14.29           | 90.00             |
| 0.03      | 0.28                      | 0.034                       | 20.00           | 90.29             |
| 0.04      | 0.27                      | 0.033                       | 22.86           | 90.57             |
| 0.05      | 0.26                      | 0.033                       | 25.71           | 90.57             |
| 0.06      | 0.24                      | 0.033                       | 31.43           | 90.57             |
| 0.07      | 0.23                      | 0.034                       | 34.29           | 90.29             |
| 0.08      | 0.22                      | 0.013                       | 37.14           | 96.29             |
| 0.09      | 0.21                      | 0.038                       | 40.00           | 89.14             |
| 0.10      | 0.20                      | 0.038                       | 42.86           | 89.14             |

**Figure 1.** Comparison of pod and leaves with dosages by varying dosage and time.
Table 2. Comparison of pod and leaves with different contact time.

| Time (minutes) | Concentration (mg/l) Pods | Concentration (mg/l) leaves |
|----------------|---------------------------|-----------------------------|
| 5              | 0.250                     | 0.034                       |
| 10             | 0.250                     | 0.016                       |
| 15             | 0.260                     | 0.013                       |
| 20             | 0.250                     | 0.013                       |
| 25             | 0.230                     | 0.013                       |
| 30             | 0.200                     | 0.013                       |

3.2 Characteristics Study Before and After Treatment

3.2.1 Energy Dispersive X ray (EDX)

It is observed from table 3 and 4, the formation of carbon is 33% in leaves whereas in pods the formation is 20.3%. It is found the formation of carbon in leaves is 15% more than pod. This causes increases the removal efficiency. From this it is evident that more carbon content increases the removal of pollutants. The carbon content can be enhanced additionally by adopting physic chemical treatment like acid or alkali based treatment. The amount of oxygen in pods is 47.1% whereas in leave is 40.3%. From this the least value of oxygen is found in leaves. As per [12], least amount of oxygen enhances the attachment of pollutants on the surface of adsorbent. This result corroborates our findings. Figure 2 and 3 shows the elemental composition present in pods and leaves. In both leaves and pods the following elements such as sodium, potassium, magnesium, silica, phosphorous, calcium is present. In addition to that leaves contain nitrogen, aluminium and sulphur whereas in pods titanium and copper is present.

![EDAX image for pod adsorbent](image-url)
3.2.2. Scanning Electron Microscope (SEM)

The surface characterisation of adsorbent before and after adsorption has been done for identifying the removal of adsorbate in leaves and pods. Figure 4, the shape of the pores is rounded in nature and it is formed as very fine powder. It is observed that several fine pores are developed in Moringa oleifera

![EDAX image for Leaf adsorbent](image)

**Figure 3.** EDAX image for Leaf adsorbent

**Table 3.** EDX results of Moringa oleifera leaves.

| Element | Weight | Atomic % | Error % | K ratio |
|---------|--------|-----------|---------|---------|
| C       | 33     | 44.0      | 8.0     | 0.1314  |
| N       | 4.1    | 4.7       | 18.7    | 0.0044  |
| O       | 40.3   | 40.3      | 10.2    | 0.0649  |
| Na      | 2.2    | 1.6       | 10.8    | 0.0075  |
| Mg      | 3.3    | 2.2       | 7.6     | 0.0158  |
| Al      | 0.3    | 0.2       | 16.9    | 0.0020  |
| Si      | 0.6    | 0.3       | 9.8     | 0.0043  |
| P       | 2.1    | 1.1       | 5.4     | 0.0157  |
| S       | 0.5    | 0.3       | 13.0    | 0.0044  |
| K       | 1.5    | 0.6       | 8.3     | 0.0132  |
| Ca      | 12.0   | 4.8       | 2.2     | 0.1053  |

**Table 4.** EDX results of Moringa oleifera pod.

| Element | Weight | Atomic % | Error % | K ratio |
|---------|--------|-----------|---------|---------|
| C       | 20.3   | 29.9      | 9.5     | 0.0613  |
| O       | 47.1   | 52.0      | 9.6     | 0.0944  |
| Na      | 1.6    | 1.2       | 12.4    | 0.0053  |
| Mg      | 8.5    | 6.2       | 6.9     | 0.0408  |
| Si      | 0.7    | 0.4       | 11.2    | 0.0047  |
| P       | 5.3    | 3.0       | 4.2     | 0.0390  |
| K       | 5.2    | 2.4       | 3.1     | 0.0456  |
| Ca      | 10.4   | 4.6       | 2.7     | 0.0899  |
| Ti      | 0.3    | 0.1       | 27.5    | 0.0021  |
| Cu      | 0.6    | 0.2       | 23.9    | 0.0048  |
leaves surface while preparation of char particle. Also it is evident from figure 4 the dye molecule is attached on the activated surface area of leaves. Before adsorption, the pores present in the adsorbent are fully open, after the adsorption the pores are occupied by the pollutants and transforms into bulk particles. The surface area of MO leaves is modified after adsorption of dye molecules. This supports the results of [12]. In figure 5, it is found that the elongated teeth shaped porous structure was formed and the particle sizes are litter bigger and uneven in Moringa oleifera pods. The particular temperature (350°C), leaves can be break down into very fine powder whereas pods needs to raise the temperature for converting into very fine powder. The reason could be the texture and structure is totally different for pods and leaves. So when we are selecting any adsorbent the preparation procedure of adsorbent is very important. Also it is recommended to prepare different methodology of preparation for pods, leaves, seeds, barks etc for the same plant. Depending upon the nature, the physico chemical treatment like KOH treatment is recommended to increase the porosity of structure subsequently the uptake capacity of adsorbent can be increased [13].

3.3 Development of isotherm
The isotherm results are predicted for the desired effluent concentration necessary adsorbent dosage can be predicted for methylene blue removal for both pod and leaves of Moringa oleifera. Constants which are used for developing isotherm are given in table 5. The correlation coefficient ($R^2$) is used for identification of suitable isotherm for pod and leaves. Freundlich isotherm is well suited for prediction for pod ($R^2=0.9226$) and the Langmuir isotherm is well suited for leaves and the correlation value ($R^2$) is 0.9691.
Table 5. Constant values for Isotherm.

| Parts of Moringa Oleifera | Constants of Freundlich | Constants of Langmuir |
|---------------------------|-------------------------|-----------------------|
|                           | $K_f$   | $n$   | $R^2$ | $\frac{1}{Q_o}$ | $b$   | $R^2$ |
| Pod                       | 0.1389 | 1.4268 | 0.9226 | 6.4371 | -1.2739 | 0.8006 |
| Leaves                    | 0.8225 | 0.6539 | 0.8578 | 0.3438 | -0.0009 | 0.9691 |

4. Conclusion
In this study an attempt is made to assess the effectiveness of the adsorption on dyes by adding activated carbon made from the leaves and pods of Moringa oleifera. The following conclusions are arrived from this study:

i. The leaves adsorbent removes almost 96% at the optimum dosage of 0.08g/100ml at a time period of 15 minutes, whereas in pod the optimum dosage is 0.1g/100ml at a time period of 30 minutes the removal is nearly 45%. The prepared activated leaf carbon is more effective in removing organic dye particles than pod.

ii. The morphological character studies carried out for the adsorbent by various techniques (SEM and EDX). It proves the adsorbent efficiency increases, if the amount of carbon content is more in adsorbent. Similarly lesser oxygen level increasing the adsorption of pollutants on the surface of adsorbent. SEM results clearly showed the several fine pores are developed in leaf adsorbent. The particles with very fine pores cause more uptake of pollutants.

iii. The adsorption isotherms are developed for MO leaves and pod for Methylene Blue dye solution and identified as the most suitable isotherms with correlation factor for both pod and leaves.

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