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Removal of Organic Pollutants from Textile Mill Effluent using *Azadirachta Indica* Powder

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Abstract

Due to stringent environmental regulations and increased awareness about the environment, effluent treatment has always been a key aspect of research. Textile industry is one of the oldest and technologically advanced complex industrial sectors employing variety of chemicals and large amount of fresh water consumption. These effluents contain substantial amount of organic pollutants and suspended impurities in varying compositions and hence cannot be discharged directly into the environment. The current research work focused on the utilization of *Azadirachta Indica* powder as a natural adsorbent for the effective removal of organic and suspended pollutants from textile mill effluent. A series of batch experimental studies were performed by varying the pH of textile mill effluent, stirring time, stirring speed and dosage of *Azadirachta Indica* powder. Chemical Oxygen Demand (COD), Total Dissolved Solids (TDS), Total Suspended Solids (TSS), Dissolved Oxygen (DO), conductivity, and turbidity were estimated to study the performance of *Azadirachta Indica* powder in the removal of pollutants. The characterizations of *Azadirachta Indica* powder before and after treatment are performed using Scanning Electron Microscopy (SEM), Energy Dispersive X-ray (EDX), X-ray Diffraction (XRD) and Fourier Transform Infrared Spectroscopy (FTIR). The best processing conditions for the effective removal of pollutants from textile mill effluent was obtained at an effluent solution pH 4.0, stirring time 90 min, stirring speed 150 rpm with 1.0 g of *Azadirachta Indica* powder. The surface morphological structure of *Azadirachta Indica* powder after batch treatment using scanning electron microscopy indicated that the pollutants are accumulated in the form of clusters on the adsorbent surface. The FTIR spectral analysis indicates the chemical interaction between adsorbent powder and organic pollutants illustrate the characteristic peaks. The study demonstrates that the *Azadirachta indica* is a promising type of biomass for the effective removal of pollutants from textile mill effluent.

Keywords: Adsorbent, *Azadirachta indica*, Bio sorbent, Chemical oxygen demand, Textile industry effluent

Introduction

Population growth, rapid urbanization and fast development in industrialization have resulted in generation of huge quantities of wastewater. Textile industry is one of the large-scale industries consumes enormous amount of fresh water and discharges almost equal quantity of wastewater [1]. Owing to the effects of water shortage and stringent environmental regulations, the cost of fresh water consumption has increased worldwide. Textile industry uses very strong chemicals during the dyeing process and releases contaminated water into the environment. Disposal of polluted water from various processing stages of textile industry results in major economic and societal impact [2]. The waste water discharged from textile mills composed of biodegradable and non-biodegradable chemicals such as dyes, dispersants, leveling agents etc. which may cause serious health and environmental problems. To meet these challenges, sustainable treatment methods need to be adopted to prevent harmful pollution and protect the public health and surrounding ecosystems [3]. Therefore, the elimination of pollutants from textile mill effluent is crucial prior to their disposal. Physical, chemical, biological, and adsorption techniques are the common treatment methods employed to remove pollutants from textile industrial wastewater. The textile mill effluent can alter the physicochemical and biological nature of the receiving water bodies by intensifying the biochemical oxygen demand (BOD), COD, TDS, and TSS as well as changes the pH with penetrating colors to water bodies [4].

Adsorption is considered as a cost effective and environmentally friendly technique [5-10] for the removal of pollutants from waste water. However, most of the treatment methods are either energy intensive process or luxurious. Activated carbon adsorption is extensively used as a conventional treatment process for the removal of toxic chemicals from industrial wastewater, but it has a limitation of high initial investment and regeneration cost, and the process releases huge quantity of sludge, which requires additional treatment steps for the safe disposal [11-14]. Therefore, it is essential to choose an efficient, cost effective and natural material for the treatment of textile mill effluent in an eco-friendly way. Wide range of materials including banana peels, eggshells, orange peels, coconut husk, surface modified straw and fiber [15-19] etc are used in the treatment processes. Neem leaf powder, a natural bio sorbent has been widely used in the removal of heavy metals from industrial effluents [20,21]. The usage of microorganisms (bacteria, fungi, and algae) and plants for the removal of azo dyes from textile wastewater is an attractive option over the physico-chemical methods [22]. Neem (*Azadirachta Indica*) tree is a naturally abundant evergreen tree find applications in medicine as antibacterial, antifungal, contraceptive, sedative, and antiviral, known as wonder leaf [23-25].

In this research, the potential of *Azadirachta indica* powder as a bio sorbent for the treatment of textile mill effluent using batch adsorption studies in a cost effective and ecofriendly method was studied. To the best of our knowledge, no major research has been reported on the use of *Azadirachta indica* powder for the effective treatment of real time textile industry effluent as most of the studies are carried out using a simulated dye solution. Neem trees are cultivated on a large scale in Oman where they are of significant agronomic importance. In this work, *Azadirachta indica* powder was used as a low-cost adsorbent to remove organics present in the Oman textile mill effluent.

Materials and methods

Raw materials

The effluent samples were provided by Oman textile mill company, Oman and preserved at 4 °C to avoid any bacterial contamination. The chemicals and reagents were procured from Sigma Aldrich India and used as supplied. The COD digester (AQ 400, thermo scientific Orion COD125), turbidity meter (WTW Turb 550) and pH meter (JENWAY 3520) were used in the characterization and analysis of the effluent samples before and after treatment. Water analysis kit was employed for the measurement of DO, TSS and TDS. The methodology used in the removal of pollutants is bio sorption technique.

The morphological characterization of samples was carried out using high resolution Field Emission Scanning Electron Microscope (FSEM JEOL JSM-7600F). The composition analysis and structural characterization of the samples were performed using X-ray diffractometer (Rigaku, and Mini Flex 600), and surface functional groups present in the samples are identified using Fourier-Transform Infrared Spectroscopy (FTIR - Frontier is Perkin Elmer’s). All batch experiments were performed in triplicate and the average of 3 readings are reported as final value. The pollutant removal efficiency was evaluated using Eq. (1) [26, 27];

$$\text{Percentage pollutant removal efficiency} = \frac{A-B}{A} \times 100 \tag{1}$$

where A and B are the initial and final concentrations of the samples in mg/L, respectively.

Preparation of bio sorbent

The *Azadirachta indica* powder was prepared by drying the neem leaves under sunshade for 5 days. The dried samples were ground into fine powder and the dried samples were sieved to get a desired size of 75 µm and stored in an airtight container for characterization and effluent treatment. The physicochemical characteristics of textile mill effluent samples before and after treatment processes are examined as per APHA standards [28]. The characteristics of the textile industry effluent before treatment are represented in **Table 1**.

Table 1 Characteristics of textile industry effluent before treatment.

Parameters	Value
pH	10.0
Conductivity, µS	6412
Turbidity, NTU	48
COD, mg/L	377
TDS, mg/L	4402
TSS, mg/L	22.7
DO, mg/L	7.35

Batch adsorption studies

A batch reactor of 500 mL capacity was employed to perform the experimental studies. The parameters under investigation were the effluent solution pH, speed of agitation, processing time, and bio sorbent dosage. The outlet samples from each experiment were collected at regular time intervals and the optimization of processing conditions were established. The batch adsorption study was performed at room temperature by mixing a calculated amount of *Azadirachta indica* powder with the textile effluent in a reactor at specified time to attain the equilibrium, after which the samples were centrifuged at 5,000 rpm for 5 min. The resulting supernatant was analyzed for the measurement of COD, TDS, TSS, DO and turbidity. The percentage removal efficiency of each parameter was determined using the Eq. (1).

Influence of pH variation

In order to determine the optimum pH for the removal of pollutants from textile effluent, experiments were performed at different pH values ranging from 2.0 to 14.0. The pH of the effluent solution was adjusted using 0.1 N NaOH and 0.1 N HCl. The experiment was conducted by mixing 1.0 g of *Azadirachta indica* powder with 250 mL of textile mill effluent with a mixing speed of 100 rpm. After the experiment, the samples were centrifuged at a rotation speed of 5,000 rpm for 5 min and the supernatant was analyzed for the measurement of COD, TDS, TSS, DO, conductivity and turbidity.

Influence of stirring time variation

Stirring time is an important factor determining the pollutant removal efficiency. The influence of change in stirring time on adsorption efficiency of *Azadirachta indica* powder at room temperature was examined by varying the contact time from 30 to 120 min. Other parameters like dosage of *Azadirachta indica* powder, effluent solution pH, and stirring speed were kept constant.

Influence of agitation speed variation

The influence of variation of agitation speed on adsorption efficiency of *Azadirachta indica* powder was studied by altering the speed of agitation from 25 to 175 rpm keeping all other experimental parameters constant.

Influence of variation in dosage of *Azadirachta indica* powder

The dosage of *Azadirachta indica* powder is a crucial factor regulating the economics of the effluent treatment system. The percentage removal efficiency of pollutants from the effluent is generally enhanced by increasing the dosage of *Azadirachta indica* powder.

The influence of dosage of *Azadirachta indica* powder on pollutant removal was studied by changing the adsorbent dosage from 0.2 to 2.0 g by fixing all other processing parameters constant. The solution was kept in a shaker at optimized conditions of pH, contact time and stirring speed.

Results and discussion

Preparation of bio sorbent powder

The surface morphology and composition analysis of the *Azadirachta indica* powder before treatment was characterized using SEM, Energy Dispersive X-ray analysis (EDX), XRD and FTIR spectroscopic analysis. The SEM image of the fresh *Azadirachta indica* powder before treatment was captured at a magnification of 1,000× and a voltage of 15.0 kV as shown in **Figure 1**. The SEM image in

Figure 1 shows *Azadirachta indica* particles are evenly distributed and exhibited a highly porous and heterogeneous structure with varying particle size and shape.

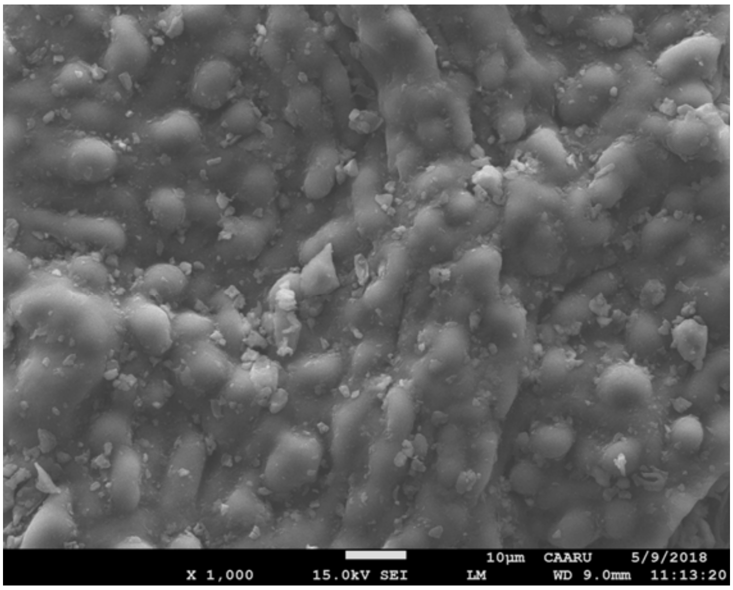

Figure 1 SEM image of *Azadirachta indica* powder at a magnification of 1,000× (before treatment).

SEM- EDS analysis

The EDX analysis of the *Azadirachta indica* powder showed in **Figure 2** displays several peaks representing carbon, oxygen, calcium, potassium, sulfur, and traces of magnesium. The data obtained from SEM-EDX analysis shown in the **Figure 2** indicates 75 % carbon, 19.8 % oxygen, and trace amounts of calcium, potassium, and magnesium.

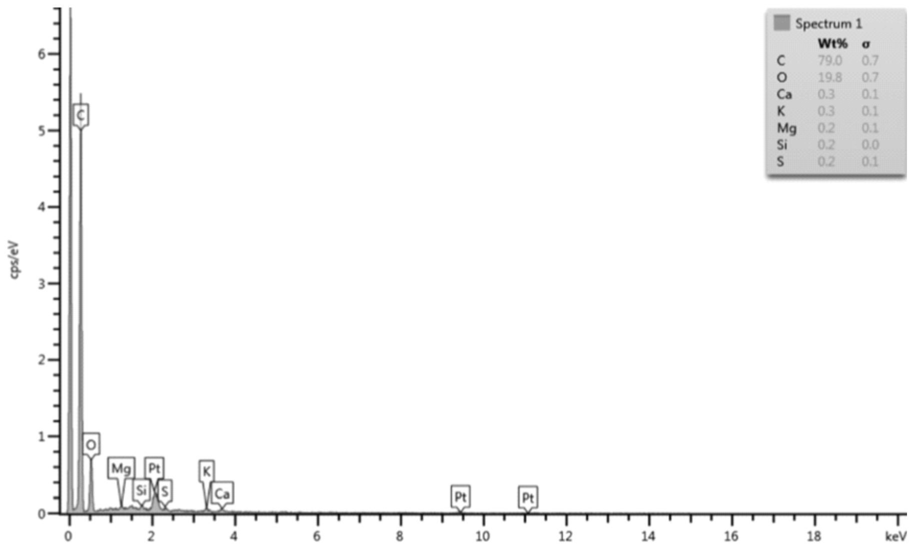

Figure 2 SEM-EDX elemental analysis of *Azadirachta indica* powder (before treatment).

Adsorption studies

Influence of effluent solution pH variation

The influences of change in effluent solution pH on pollutant removal efficiency are indicated in **Figures 3** and **4**. The maximum reduction in conductivity, turbidity and dissolved oxygen was observed at pH 2.0. As seen from **Figure 3**, an increase in effluent solution pH tends to form a drop in conductivity, turbidity, and dissolved oxygen. The influence of pH changes on the removal of TDS, TSS and COD are shown in **Figure 4**. From the **Figure 4**, the highest COD removal efficiency was observed at pH 4.0, above which there was a decreasing trend until pH 12.0. There was a surge in percentage reduction of TSS due to an increase in the effluent pH for all the ranges of values studied. The driving force for the adsorption of pollutants on the surface of *Azadirachta indica* powder is electrostatic interaction. Higher electrostatic interaction between the surface of adsorbent and pollutants resulted in higher adsorption rate. At lower pH, the hydrogen ions compete with the pollutants present in the effluent in occupying the suitable sites. The reduced percentage removal efficiency is due to the diminishing surface charge, which will slow down the effluent treatment process. The structural properties of the molecules present in the textile effluent is responsible for linking of hydroxyl groups of *Azadirachta indica* powder via nucleophilic addition or a nucleophilic substitution reaction. Such type of reaction indicates that the pH of the effluent would have a strong influence on the adsorption process.

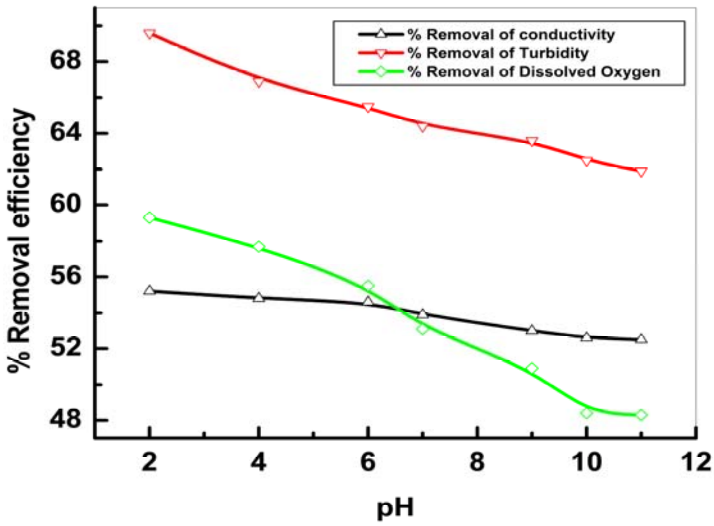

Figure 3 Effect of change in pH on the % removal of DO, conductivity and turbidity.

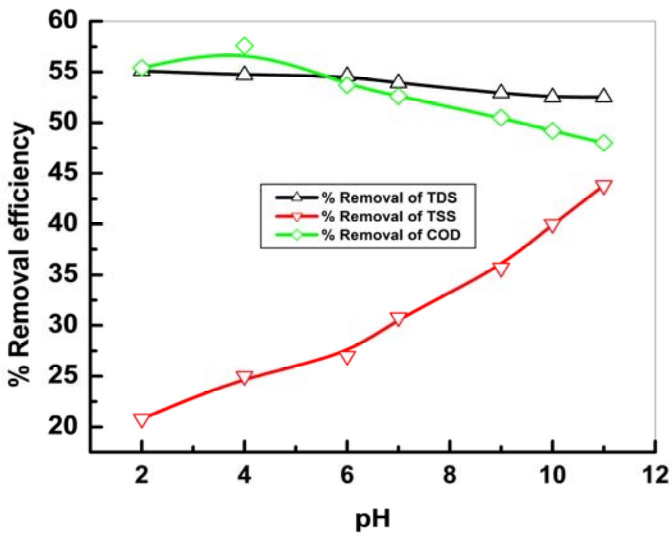

Figure 4 Effect of change in pH on the % removal of TDS, TSS, and COD.

In **Table 2**, represents the characteristics of the effluent after treatment with *Azadirachta indica powder*.

Table 2 Characteristics of textile industry effluent after treatment.

Parameters	Values
pH	7.2
Conductivity, µS	3101
Turbidity, NTU	15
COD, mg/L	212
TDS, mg/L	1456
TSS, mg/L	13
DO, mg/L	4.5

Influence of stirring time variation

Stirring time is a prominent factor determining the effectiveness of parameter reductions during the waste water treatment processes. The influence of change in stirring time on the effective removal of pollutants was attained by changing the contact time from 25 to 150 min by maintaining the other parameters same. **Figures 5** and **6** displays the graphical representation of the percentage removal efficiency of pollutants with changes in contact time. With increase in stirring time, the deposition of pollutants on the active sites of adsorbent will be faster and more effective. The percentage reduction in COD was increased to 68 % in the range of contact time studied between 25 to 150 min as illustrated in **Figure 6**. A gradual increase in the percentage removal efficiency in the conductivity and dissolved oxygen with time was observed from the **Figure 5**. The experimental outcome demonstrates that an increase in agitation time results a surge in the conductivity and dissolved oxygen. However, the turbidity decreased with increase in agitation time with a maximum turbidity reduction corresponding to 25 min of contact time.

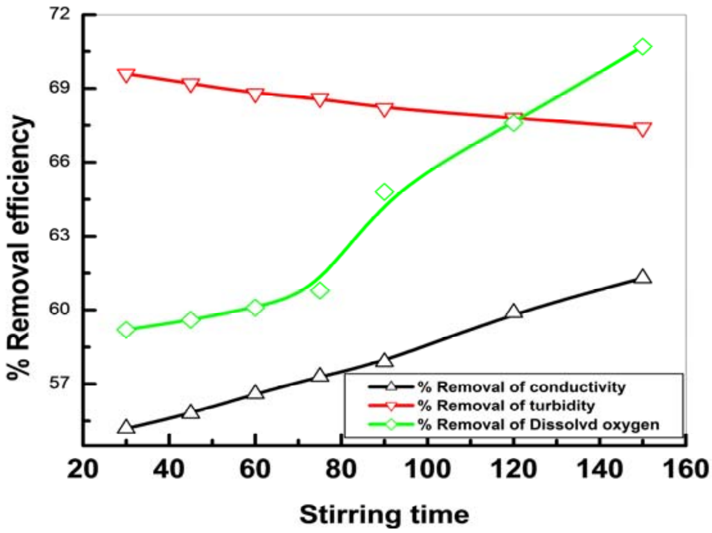

Figure 5 Effect of change in stirring time on the % removal of conductivity, turbidity, and dissolved oxygen.

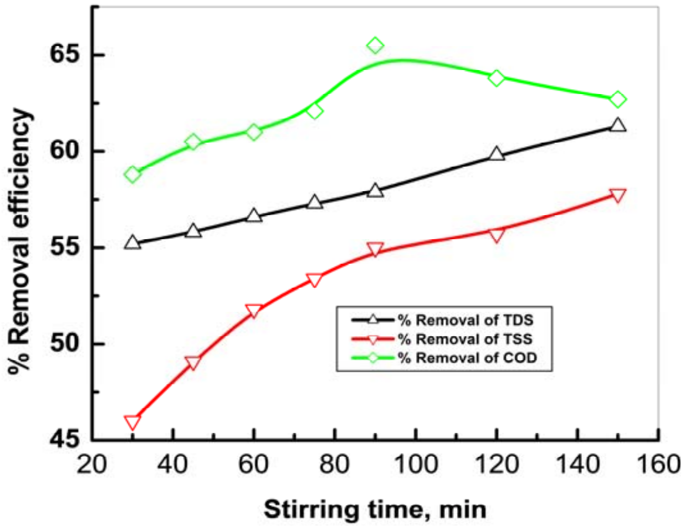

Figure 6 Effect of change in stirring time on the % removal of TDS, TSS, and COD.

As seen in the **Figure 6**, the maximum reduction in TDS, TSS and COD are achieved at a stirring time of 150 min. The improved rate of adsorption is due to the attraction of pollutants towards active functional groups available on the bio sorbent surface, which led to a stronger surface binding. As the active sites are completely occupied by the adsorbate, the adsorption is controlled by the rate at which the pollutants are transported from exterior to interior sites.

Influence of stirring speed variation

The influence of change in stirring speed with percentage reduction of COD, TDS, TSS, DO, turbidity and conductivity are studied by altering the agitation speed between 20 and 180 rpm by fixing all other parameters constant. The percentage reduction in parameters at various stirring speed is illustrated in **Figures 7** and **8**. As observed from the Figures, an increase in stirring speed results an escalation in percentage removal of both conductivity and dissolved oxygen, however there was a dip in the turbidity with enhanced stirring speed. The percentage reduction in TSS shows a direct influence on the stirring speed from 20 to 180 rpm. In addition, the highest percentage reduction in COD was observed at a stirring speed of 160 rpm. However, TDS displays a decreasing trend in the range of stirring speed between 20 and 180 rpm. It was established that the rate of mass transfer increased with increase in stirring speed and hence resulted in greater interaction between the pollutants and adsorbent surface.

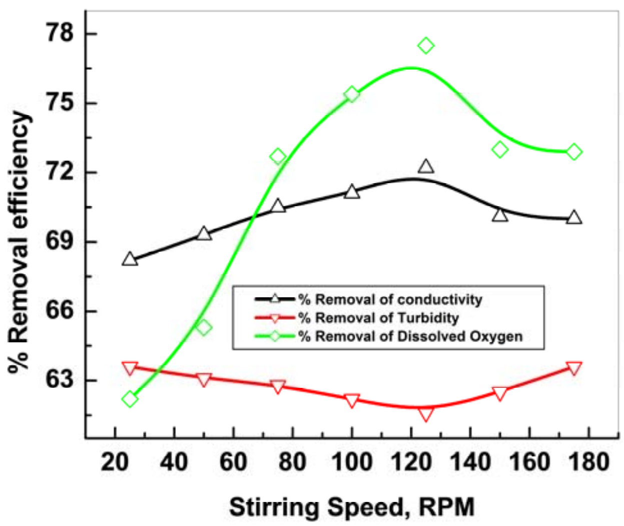

Figure 7 Effect of change in stirring speed on the % removal of conductivity, turbidity, and dissolved oxygen.

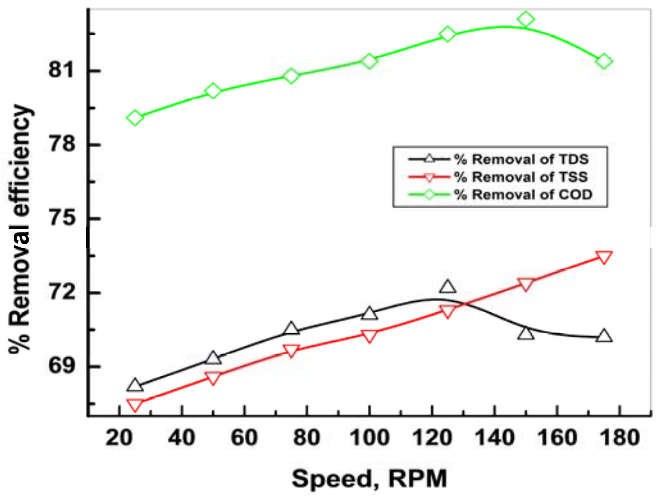

Figure 8 Effect of change in stirring speed on the % removal of TDS, TSS, and COD.

Influence of variation in the dosage of Azadirachta indica powder

Loading of the *Azadirachta indica* powder is a critical factor determining the economics of the effluent treatment process. The removal efficiency of organics generally enhanced with increasing the quantity of *Azadirachta indica* powder. It is anticipated that the increased dosage of *Azadirachta indica* powder will lead to greater accessibility of active sites on the surface of the powder and hence more surface area available for adsorption. The dependence of percentage reduction in parameters on the amount of adsorbent was studied by varying the amount from 0.2 to 2.0 g and all other parameters (solution pH, stirring time and stirring speed) kept constant. **Figures 9** and **10** represent the graphical representation of the percentage removal efficiency at varying amounts of *Azadirachta indica* powder. The percentage reduction of COD increased to 78 % at an optimum dosage of 1.2 g/100 mL of the effluent for the effective removal of organic pollutants present in wastewater samples. **Figure 9** displays the variation of adsorbent dosage with percentage removal of conductivity, turbidity, and dissolved oxygen. The graph indicates an increase in bio sorbent dosage with increased percentage removal of conductivity and dissolved oxygen. However, the turbidity decreased with increase in the dosages. The percentage removal efficiency of TDS, TSS and COD with different dosages of *Azadirachta indica* powder is shown in **Figure 10**. An increase in the bio sorbent dosage increased the percentage removal efficiency of TDS, TSS and COD. Hence the dosage of adsorbent plays an important role in the parameter reductions. With an increase in the amount of *Azadirachta indica* powder resulted in the number of active sites on the surface of adsorbent powder and hence leads to the enhancement of pollutant removal efficiency. The results showed that, as the amount of *Azadirachta indica* powder increases, the percentage of pollutants deposited on the surface of the adsorbents also increased. This trend could be attributed to the availability and accessibility of more adsorption sites and more surface area for the pollutant attachment [29-31]. It was observed that, as the amount of *Azadirachta indica* powder increased beyond 1.2 g, there seemed to be no more significant increase in the COD removal efficiency of bio sorbent powder [32]. This could be due to the unavailability of empty sites on the surface of the bio sorbent. This favors the possible application of neem leaves powder in the removal of pollutants from effluent at a minimum dosage.

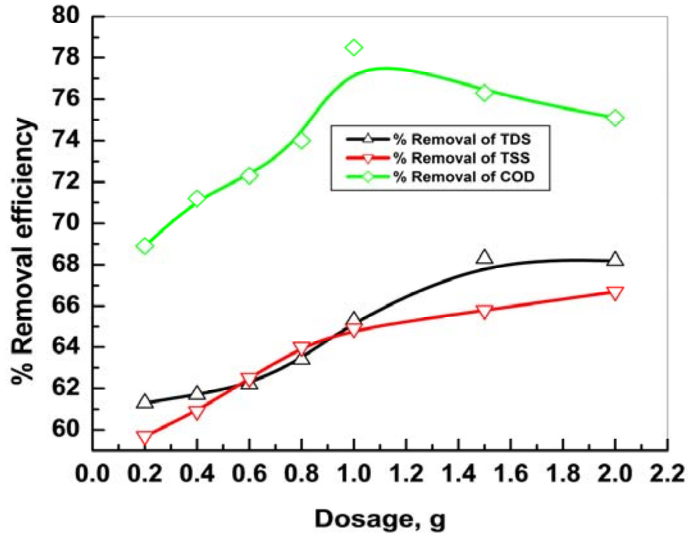

Figure 9 Effect of change in dosage of *Azadirachta indica* powder on the % removal of conductivity, turbidity, and dissolved oxygen.

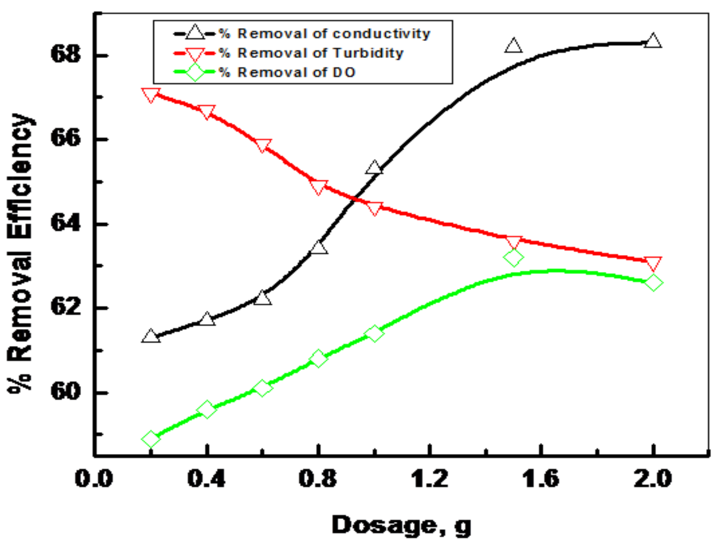

Figure 10 Effect of change in dosage of *Azadirachta indica* powder on the % removal of TDS, TSS, COD.

The functional groups present on the surface of *Azadirachta indica* powder, before adsorption was identified by using FTIR spectroscopy as shown in **Figure 11**. The infrared spectra indicated the actual bond stretching and different functional groups available on the surface of sample. **Figure 11** represents the indication of the type of functional groups present in the *Azadirachta indica* powder sample before treatment. Characteristic peaks including O-H stretching of water 3419 cm^{-1}, and O-H deformation of water 1436 were observed in the infrared spectrum of *Azadirachta indica*. The stretching vibrations of hydroxyl groups at the surface of adsorbent gave the characteristic double peaks at 3392 and 3225 cm^{-1}. The bands observed at 1642 cm^{-1} indicates the stretching vibrations of the carbonyl functional group in ketones (=CO), Aldehydes (-CHO) and carboxylic acids (-COOH). The second absorption at 1536 cm^{-1} corresponds to the amide I ($-CONH_2$) band. These functional groups contribute the interaction properties of the *Azadirachta indica* powder and the pollutants. The FTIR spectra of the *Azadirachta indica* powder surface exhibited the presence of -OH, $-NH_2$, > C=N-, =C-C=, =C-N < and =C-0-, > C = 0, > C=C C=S groups. These functional groups are expected to control the interaction between *Azadirachta indica* powder and the pollutants.

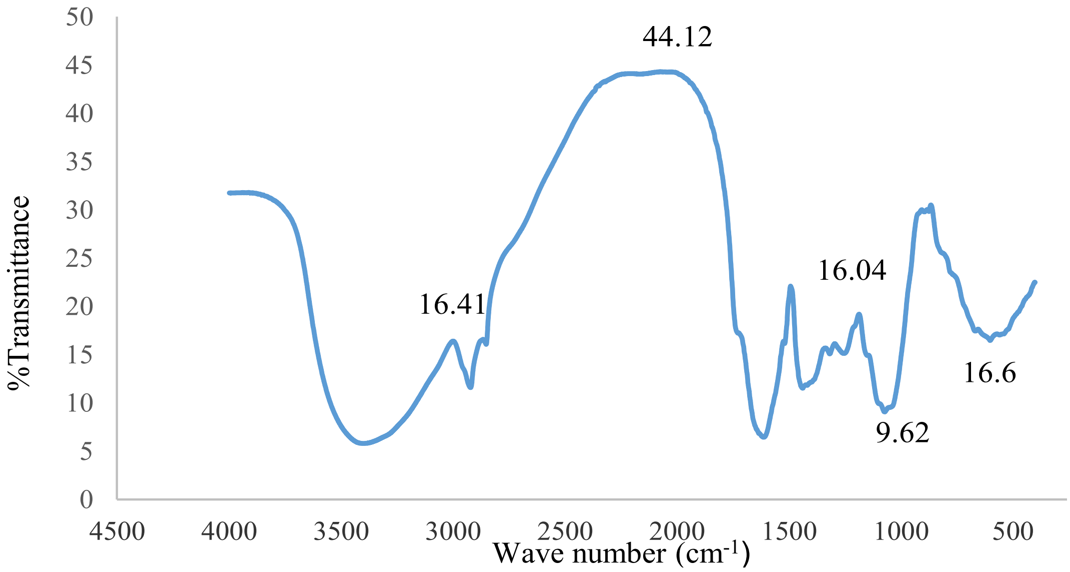

Figure 11 FTIR spectra of *Azadirachta indica* powder sample before treatment

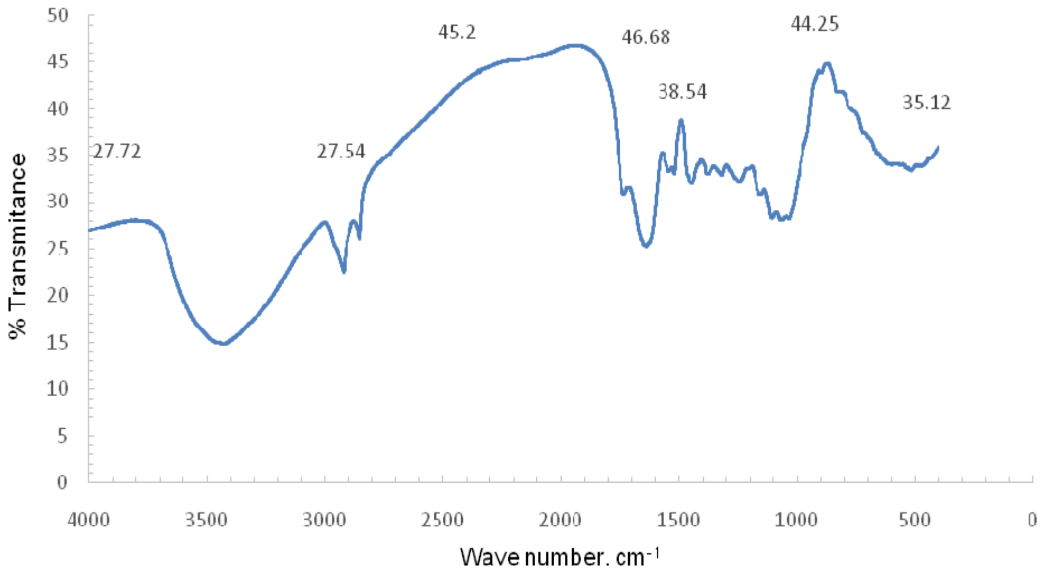

Figure 12 FTIR spectra of *Azadirachta indica* powder sample after treatment.

The FTIR spectrum shown in **Figure 12** illustrates the existence of niacin, proline, glutamic acid, aspartic acid, glutamine, tyrosine, and alanine in *Azadirachta indica* which contribute to the functional group and surface charge [33,34]. The shift in some of the peaks illustrated in **Figure 12** are due to the transformation in the vibration frequencies after effluent treatment and the chemical interaction taking place between the *Azadirachta indica* powder and the pollutants [35]. **Table 3** illustrates a comparison of the pollutant removal efficiencies of neem leaf powder and activated charcoal.

Table 3 Comparison of the pollutant removal efficiencies of *Azadirachta indica* and activated charcoal [36].

Amount of neem leaf powder (g)	% Removal Efficiency	Activated Charcoal, (g)	% Removal Efficiency
0.1	68.00	0.1	26.00
0.2	69.00	0.2	28.00
0.3	69.80	0.3	32.00
0.4	70.80	0.4	46.00
0.5	71.00	0.5	49.00
0.6	71.80	0.6	62.00

Figure 13 shows the surface morphological characteristics of *Azadirachta indica* powder after effluent treatment. The SEM micrograph of the sample after treatment was captured at a magnification of 450X and an excitation voltage of 15kV. **Figure 13** designates that the surface of the bio sorbent is fully covered with contaminants in the form of agglomerates and the surface become very thick due to the deposition of pollutants. This demonstrates that the active sites in the *Azadirachta indica* powder play a significant role in the bio sorption process. The thick surface morphology shows the successful deposition of pollutants on the surface of *Azadirachta indica* powder. The micro structural feature from the SEM image confirms the successful deposition of pollutants on the adsorbent surface in the form of non-uniform distribution of clusters. This demonstrates that some of the functional groups present on the surface of bio sorbent were responsible for the effective removal of pollutants from bulk solution. The micrograph shown in **Figure 13** clearly indicates the masking of some of the particles by dark patches over the *Azadirachta indica* powder. The surface of *Azadirachta indica* powder after treatment appears to be irregular macropores with some expanded cavities which may allow for the diffusion of the pollutants through the macropores.

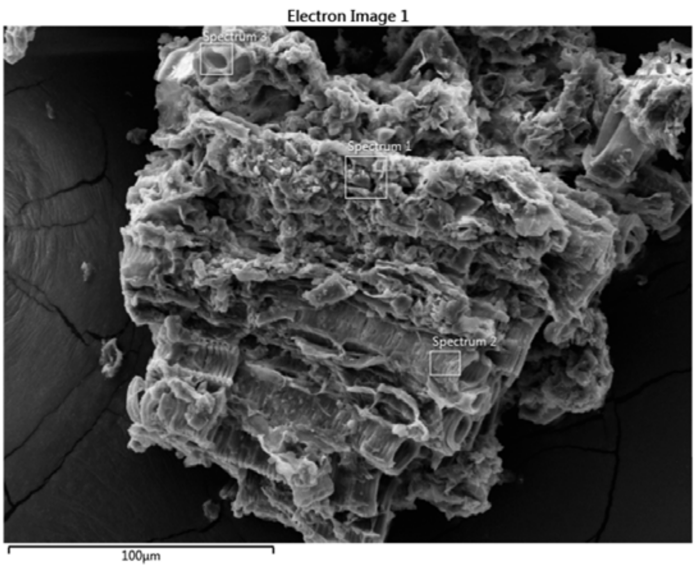

Figure 13 SEM micrograph of *Azadirachta indica* powder after treatment at a magnification of 450× and an excitation voltage of 15.0 kV

The EDX spectra of the *Azadirachta indica* powder after effluent treatment process is shown in **Figure 14** exhibiting the peaks corresponding to the elemental composition. The spectra indicate that there is a shift in position of the peaks; particularly a new peak of nitrogen is seen after bio sorption process. The elemental compositions after treatment are transformed to 58.4 % carbon, 31.5 % oxygen, 8.7 % nitrogen and the rest calcium, silicon, and sulphur. The EDX spectra indicate that the carbon content was reduced to 58.4 % whereas oxygen content was increased to 31.5after bio sorption process, which explains the effective removal of pollutants from textile mill effluent.

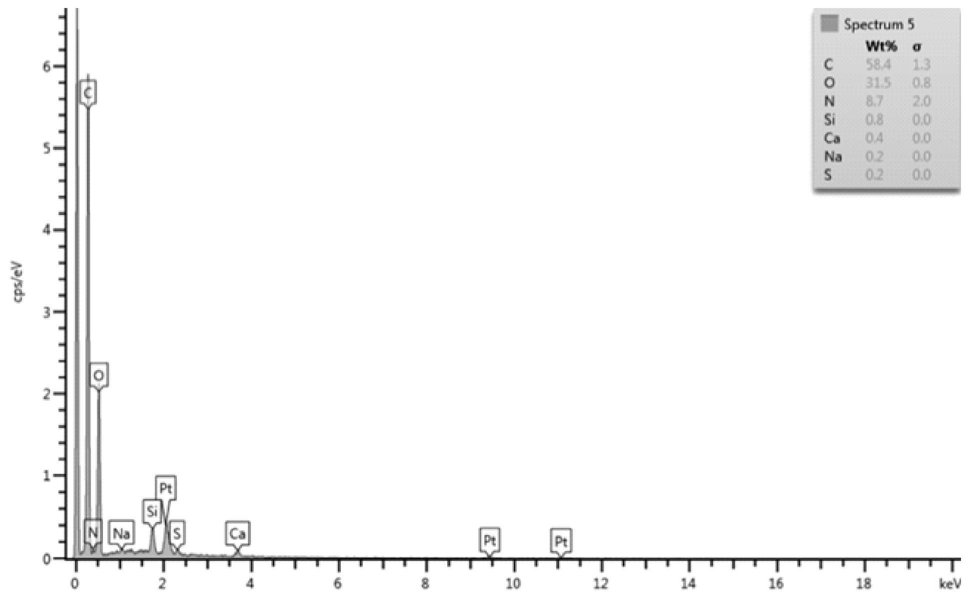

Figure 14 SEM EDX Analysis of *Azadirachta indica* powder after treatment.

The crystalline structure and phase identification of the *Azadirachta indica* powder before and after bio sorption process was analyzed using X-ray diffractometer. The difference in spectral arrangement of the sample before and after treatment is expressed in **Figures 15** and **16**. The X-ray diffraction spectrum shown in **Figure 15** indicates that no discrete mineral phase was detected. Thus, *Azadirachta indica* powder has exclusively amorphous structure, which is expected for organic materials.

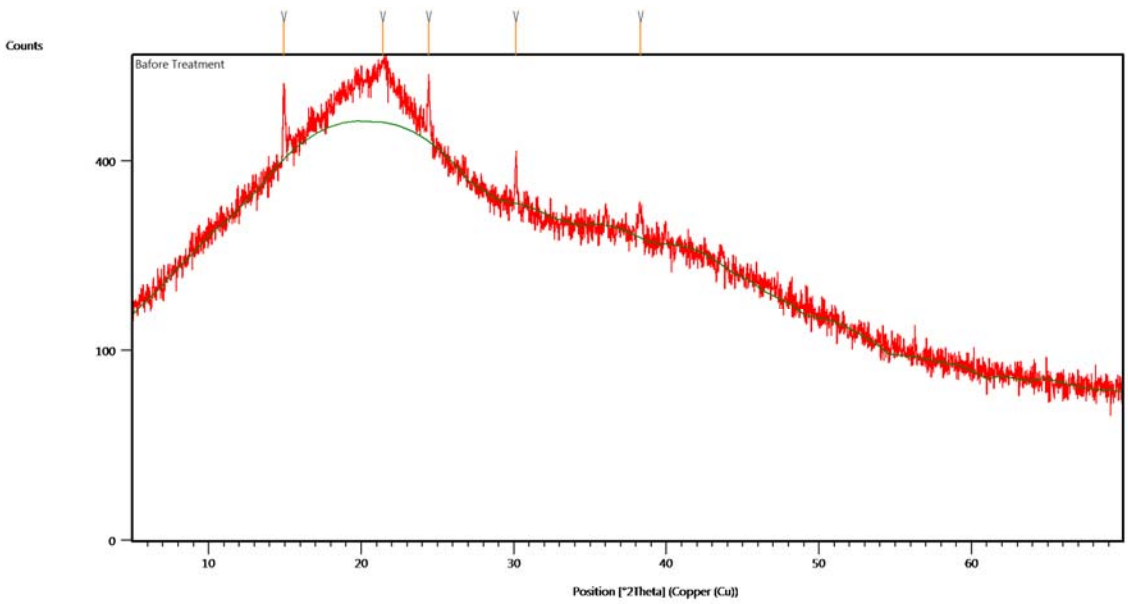

Figure 15 XRD pattern of *Azadirachta indica* powder before treatment.

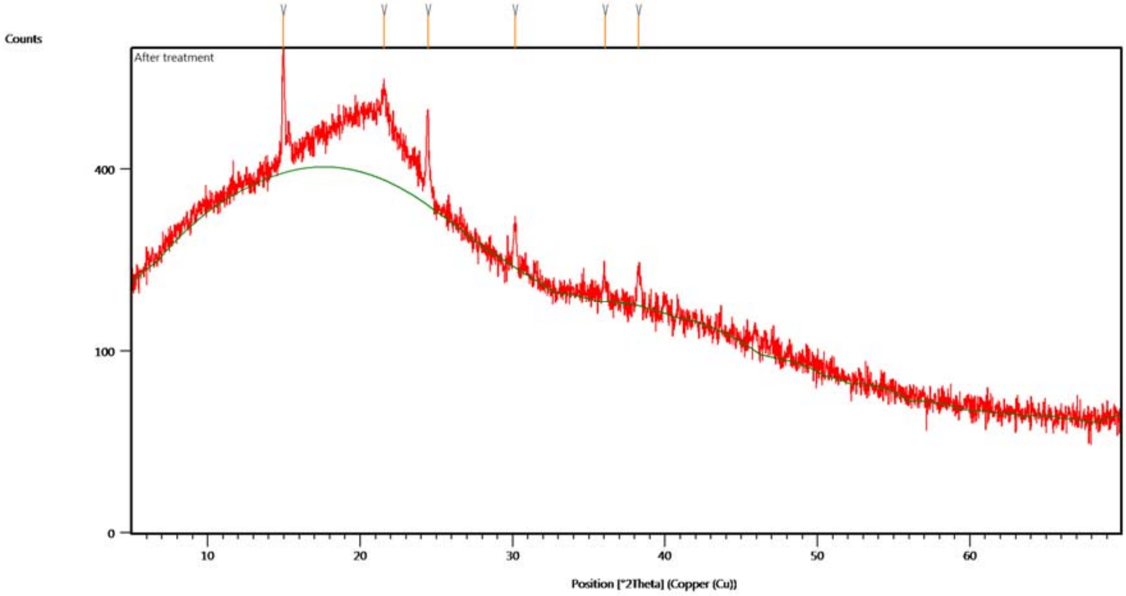

Figure 16 XRD pattern of *Azadirachta indica* powder after treatment.

Conclusions

Bio sorbents are locally available and highly effective in the removal of pollutants from industrial wastewater at an affordable cost without any harmful effects on human and living organisms. The present study focused on the preparation of *Azadirachta indica* powder as a natural and eco-friendly bio sorbent and its application in the batch treatment of textile mill effluent by varying the processing parameters. The results presented here shows that *Azadirachta indica* powder is a promising and inexpensive biosorbent for the removal of pollutants from textile mill effluent. The microstructural characterization of the adsorbent using SEM analysis displayed a highly heterogeneous surface and the X-ray diffraction spectra demonstrated a completely amorphous structure. The batch experimental study illustrates that the adsorption capacity was dependent on the processing conditions such as effluent pH, contact time, stirring speed and dosage of adsorbent. The optimum treatment conditions obtained are 4.0 pH, 90 min of stirring time, 1.0 g bio sorbent dosage, and 150 rpm as stirring speed. This eco-friendly green and cost-effective research will bring a pollution-free environment and reduce the waste generation. As a next phase of the research, the effectiveness of *Azadirachta indica* powder in the removal of pollutants from refinery and diary waste water will be studied. Also the research team is currently working on the possibility of extending the study in pharmaceutical waste water treatment for comparing the pollutant removal efficiency.

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