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Use of *Moringa oleifera* Seed as a Natural Adsorbent for Wastewater Treatment

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Abstract *Moringa oleifera* (MO) is a multipurpose, medium- or small-sized tree, from regions of northwest India and indigenous to many parts of Asia, Africa, and South America. Its pods have been employed as an inexpensive and effective sorbent for the removal of organics, and coagulant for water treatment. It is a non-toxic natural organic polymer. The main objective of this work was to use the MO seeds as a natural adsorbent for the treatment of dairy industry wastewater (DIW). The effects of agitation time, pH, MO biomass dose, and DIW concentration were evaluated. Removal efficiencies of up to 98%, for both color and turbidity, were reached using 0.2 g MO and 0.2 L of 1.0 g/L sorbate solution (DIW). The obtained results showed that MO seed keeps its adsorption power under a pH range between 5 and 8. The adsorption data was fitted to Langmuir isotherm. There was a significant uptake capacity of MO biomass, $q_{\max}$, which suggested a good affinity between DIW components and sorbent. We conclude that the MO biomass has the potential to be used in the dairy industry wastewater treatment in an efficient way and with low cost.

Keywords Dairy industry wastewater · *Moringa oleifera* · Biosorption · Natural adsorbent

1 Introduction

Turbidity and color removal is one of the important steps in a water treatment process, which is generally achieved using coagulants. Many coagulants are widely used in conventional water treatment processes, based on their chemical characteristics. These coagulants are classified into inorganic, synthetic organic polymers and natural coagulants. The two most commonly used primary coagulants are aluminum and iron (III) salts (Okuda et al. 1999). However, recent studies have pointed out several drawbacks of using aluminum salts, such as Alzheimer's disease associated with residual aluminum in treated water and production of large sludge volumes (Ndabigengesere

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and Narasiah 1998). There is also the problem of reaction of aluminum with natural alkalinity present in the water leading to a reduction of pH and low efficiency of coagulation in cold waters. To ease the problems associated with chemical coagulants, several studies have pointed out the introduction of natural coagulants produced or extracted from microorganisms, animals, or plants (Katayon et al. 2006).

Wastewater treatment methods include precipitation, coagulation/flotation, sedimentation, filtration, membrane process, electrochemical techniques, ion exchange, biological process, and chemical reactions. Each method has its own merits and limitations in applications because of their cost. Presently, there is an increasing trend to evaluate some indigenous cheaper materials for the removal of these pollutants and pesticides from aqueous solutions. A large number of cheaper materials including industrial and agricultural wastes have been used to remove different pollutants from the industrial effluents for their safe disposal into the biosphere (Akhtar et al. 2009).

Moringa oleifera (MO) is a tropical plant belonging to the family Moringaceae (Katayon et al. 2006), a single family of shrubs with 14 known species. MO is native to India but is now found throughout the tropics (Bhatia et al. 2007). It is a non-toxic natural organic polymer. The tree is generally known in the developing world as a vegetable, a medicinal plant, and a source of vegetable oil (Katayon et al. 2006). It is drought tolerant and has nutritional, medicinal, and water-cleaning attributes. Its leaves, flowers, fruits, and roots are used locally as food articles. The medicinal and therapeutic properties of this plant have led to its application as a cure for different ailments and diseases, physiological disorders, and in eastern allopathic medicine (Akhtar et al. 2007). Sludge produced by coagulation with MO is not only innocuous but also four to five times less in volume than the chemical sludge produced by alum coagulation (Ndabigengesere et al. 1995). An additional benefit of using coagulants derived from MO is that a number of useful products may be extracted from the seed. In particular, edible and other useful oils may be extracted before the coagulant is fractionated. Residual solids may be used as animal feed and fertilizer, while the shell of the seed may be activated and used as an adsorbent. The coagulant is thus obtained at extremely low or zero net cost (Ghebremichael et al. 2005).

In terms of water treatment applications, MO seeds in diverse extracted and purified forms have proved to be effective in removing suspended material. MO extracts generate lower sludge volumes in comparison with aluminum, soften hard waters, and act as effective adsorber of cadmium. If a physico-chemical treatment applied during the first stage of the wastewater treatment is effective, then the organic load on any subsequent biological treatment phase will be considerably reduced (Bhuptawat et al. 2007).

The major concern in the use of seed extracts for water treatment applications is the residual organic seed material that will be present in the finished water. MO is organic and biodegradable. If the particulates are removed and the sludge that is generated is proven to be non-hazardous by analysis, then this sludge may be used as a fertilizer and/or soil conditioner after stabilization (Bhuptawat et al. 2007).

If MO is proven to be active, safe, and inexpensive, it is possible to use it widely for drinking water and wastewater treatment. MO may become one of the cash products bringing more economic benefits for the producing countries (Okuda et al. 1999).

In the processing of milk for consumption “in natura”, operations that generate wastewater are: washing and disinfection of equipment (tanks, centrifuges, pasteurizers, homogenizers, pipes, pails, etc.), loss of packages containing milk, and loss in transportation. Depending on the type and capacity of the industry, the raw sewage generated can reach high values of chemical oxygen demand (COD).

Wastewater coming from cheese-producing industries is high in organic matter (about 40–60 g/L COD) since it generally contains discarded cheese whey as well (Gavala et al. 1999).

The use of vegetal biomass as a bio-filter for remediation of waters contaminated with pesticides or metals has been widely described in the literature over the last 10 years (Akhtar et al. 2007a, b). However, the adsorption capacity and the affinity of these bioadsorbents greatly fluctuate according to their origin. The recourse to a ubiquitous available adsorbent biopolymer with high adsorption properties toward a large variety of organic compounds should contribute to the development of these substrates (Alila and Boufi 2009).

Cellulose and lignocellulosic materials are among the most important organic polymers derived from the biomass. Typical examples of these polymers are nitrocellulose, hydroxyethylcellulose, carboxymethylcellulose, cellulose acetate, and cellulose acetobutyrate. Though the most adopted approaches for cellulose modification call upon reactions carried out in homogeneous conditions, giving rise to a new chemical backbone, the heterogeneous modification is the most appropriate procedure to enhance the sorption properties of lignocellulosic fibers toward heavy metal ions or organic pollutants (Sciban et al. 2006).

Our recent efforts have been focused on the coagulation and adsorption process with MO for water and wastewater treatment. We are now reporting an improved preparative strategy aimed at a much effective and cheaper product that can remove turbidity, color, and COD from DIW components.

MO seed has been widely studied for the extraction of an active compound, used in coagulation/flocculation processes (Gassenschmidt et al. 1995; Bhatia et al. 2007; Okuda et al. 1999) as well as for the adsorption of different compounds in biosorption processes.

Some studies used the MO as adsorbent for the removal of organic pollutants (Akhtar et al. 2007a) and metals (Sharma et al. 2006; Bhatti et al. 2007) from aqueous solutions, but no previous work has appeared concerning the removal of dairy industry wastewater (DIW) components, like proteins, lipids, and carbohydrates, using MO pods. The present communication deals with the sorption of DIW components onto cost-effective MO.

The main objectives of the present work are to investigate the sorption potential of MO for the removal of organics—DIW components from aqueous media over a wide pH range along with other parameters affecting the sorption process. Data has been analyzed by a statistical model. A kinetic study of the sorption process has also been made. This may lead to a better understanding of the sorption process and demonstrate its utility in the pre-concentration of organics from DIW samples.

2 Materials and Methods

2.1 Preparation of Synthetic Wastewater

Dairy industry wastewater (DIW) for adsorption tests was prepared by adding milk powder into tap water. Wastewaters with different concentrations were prepared in accordance with the test to be performed (0.1–2.2 g of milk powder/L of tap water). The suspension was stirred for a few minutes to complete dispersion.

2.2 Preparation of *Moringa oleifera* Biomass

The MO seeds used in this work were supplied by Federal University of Sergipe, Sergipe, Brazil. Pods biomass of MO was selected as biosorbent for the color, turbidity, and COD removal.

The biomass was cut, grounded using a food processor (Moulinex, France), and then sieved to obtain an adsorbent with homogenous known particle size. The fraction with mesh size <0.42 mm was selected for use in the adsorption tests. The seeds were not submitted to drying or any other preparation process.

2.3 Batch Biosorption Studies

In all sets of experiments, a fixed volume of wastewater solution (0.2 L) was thoroughly mixed with the desired biosorbent dose (0.15–0.25 g) at 25± 1°C at different rotational speeds for up to 60 min. Equilibrium periods of 60 min for adsorption experiments were used to ensure equilibrium conditions. This time was chosen considering the results of kinetics of organic compounds removal found in literature (Sharma et al. 2006). Different conditions of pH (5–10), contact time (5 to 60 min), and initial DIW concentration (0.1–2.2 g/L) were investigated during the study to check their influence on the process.

Adsorption tests were carried out using a Jar Test (Milan, model JT101). After a 60-min sedimentation step, an aliquot of 25 mL was sampled from the beaker and turbidity and residual color were determined. The same adsorption test was conducted, for control, with no adsorbent.

To adjust the pH of the medium, 0.1 N solutions of NaOH and HCl were used during the study.

2.4 Determination of the Turbidity, Color, and COD Contents in the Solutions

The quantification of compounds responsible for color, turbidity, and COD before and after the equilibrium was performed by different methods. The removal efficiency or percent adsorption was calculated according to Eq. (1), where Ci and Ce are the initial and equilibrium values of the evaluated parameters, expressed in NTU for turbidity, µH (mgPt-Co/L) for apparent color, and mgO_2/L for COD.

$$\text{Removal efficiency} = \frac{(Ci - Ce)}{Ci} \times 100 \tag{1}$$

2.5 Kinetics of Adsorption

The initial (Ci) and equilibrium (Ce) amounts of compounds adsorbed at various time intervals were determined and the components uptake qe was calculated from the mass balance, by means of Eq. (2). qe is the equilibrium adsorption capacity for turbidity (NTU × L/g) or color (µH × L/g). V is the volume of solution (L) and w is the amount of dry sorbent (g).

$$qe = \frac{(Ci - Ce)V}{1{,}000w} \tag{2}$$

Each experiment was conducted in triplicate to ensure the reproducibility of results. Every data represents the average of three independent experiments along with standard deviations at 95% confidence. Statistical analyses were performed using the statistical functions of Microsoft Excel version Office XP (Microsoft Corporation, USA).

2.6 Analytical Methods

All analytical methods followed the Standard Methods (APHA 1995). Turbidity measurements were conducted using a turbidimeter (HACH 2100P). The pH measurements were made on a digital pH meter (Digimed DM-2). Apparent color measurements were conducted using HACH DR/2010—Method 8025. The COD values were determined using HACH DR/2010—Method 10129.

3 Results and Discussion

The application of MO seeds in DIW treatment has not yet been reported. Consequently, there is no way to compare these results. It must be highlighted that no chemical or thermal treatment was made on MO seeds. This procedure makes the adsorption process simpler and more applicable.

Study of MO as a natural adsorbent for wastewater treatment is still recent, particularly when related to DIW. For this reason, it is difficult to discuss and compare the results with reports from other researchers. Moreover, the reported results (Gassenschmidt et al. 1995; Okuda et al. 2001; Bhuptawat et al. 2007) usually present disagreements about the variables used in the process with MO. Another point to consider is the fact that natural compounds may present variations in their composition, which interfere in its adsorption power. All those factors should be considered when evaluating the obtained results.

The indicative values of the physical and chemical parameters for the synthetic wastewater prepared in laboratory are presented in Table 1.

3.1 Influence of Initial pH

The equilibrium turbidity, color, and COD uptake of MO from DIW solutions (1 g/L) at various pH values are shown in Fig. 1.

MO pods contain cellulose, hemicellulose, lignin, and crude fiber. Its matrix network contains carboxylic, fiber carbonaceous, and amino functional groups. These functional groups may be dissociated at different pH values and consequently take part in the adsorption process. Therefore, pH may influence the adsorption of components onto MO (Akhtar et al. 2007a).

For the pH range of 5–8, a reduction of around 98% for turbidity and 95% for color was reached. For COD, percent reduction increased with an increase in pH, from 37% at pH=5 to 50% at pH=8, decreasing at pH values higher than 8.

Table 1 Indicative values of the parameters evaluated from a synthetic DIW

Parameter	Indicative value
Turbidity (NTU)	856
Color (µH)	4,006
COD (mgO_2/L)	1,299
pH	7.2

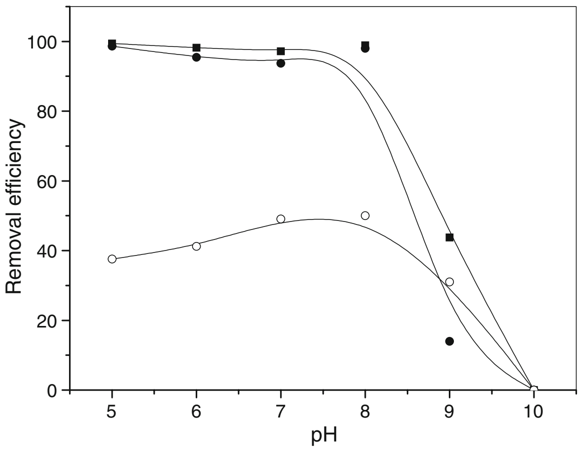

Fig. 1 Influence of pH in the range between 5 and 10 on percent reduction of color (●), turbidity (■), and COD (○) using 0.2 g MO and 0.2 L of 1 g/L sorbate solution (DIW)

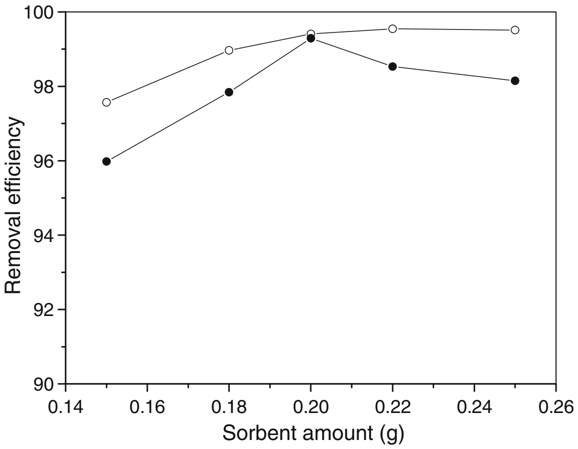

Fig. 3 Color (●) and turbidity (○) removal efficiency as a function of MO amount using 0.2 L of 1 g/L sorbate solution and an agitation time of 60 min at pH 7.2 and 25°C

This may be attributed to the presence of more hydrogen ions at lower pH, resulting in an increased uptake of DIW components by sorbent surface. On the other hand, the presence of hydroxyl ions at higher pH may result in the suppression of adsorption for all the DIW components onto MO (Bhatti et al. 2007).

It appears that there is no significant variation in removal efficiency of both color and turbidity in the pH range 5–8. However, when the pH was increased above 8, the MO adsorption power was affected significantly. Turbidity, color, and COD removal efficiencies decreased to 43%, 13%, and 31%, respectively, at pH 9. There was no significant change in color, turbidity, or COD values after the adsorption performed at pH=10. It seems that the MO adsorbent completely lost its adsorption power.

Preliminary studies on the active ingredients of *M. oleifera* as a coagulant have suggested that the active components are cationic peptides of relatively low molecular weight and isoelectric pH value of 10 (Bhuptawat et al. 2007). This could explain the lower removal efficiencies reached for turbidity, color, and COD at pH=10, since at this pH a decrease in solubility of the proteins present in MO occurs. The cationic peptides cited as responsible for the MO coagulation power may also be responsible for the adsorption of organic compounds on the surface of MO adsorbent. The fact that the seeds were not

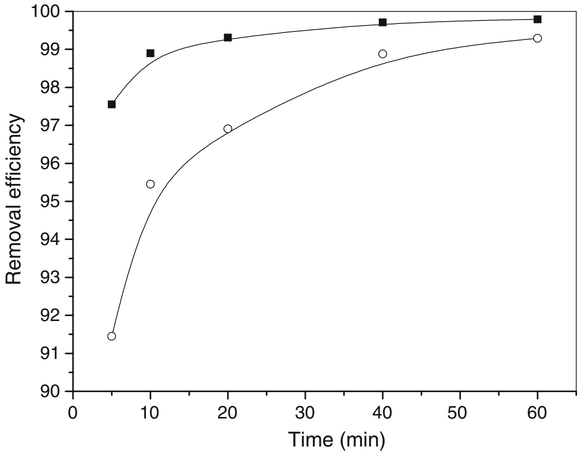

Fig. 2 Effect of agitation time from 5 to 60 min on turbidity (■) and color (○) removal efficiency from DIW, using 0.2 g MO and 0.2 L of 1.0 g/L sorbate solution at pH 7.2 and 25°C

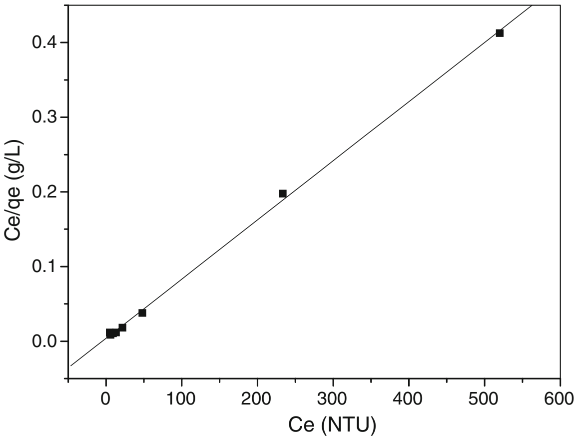

Fig. 4 Linearized Langmuir isotherm plot for biosorption of DIW components (yielding DIW turbidity) by MO biomass. (■) Experimental data; (—) fitting of model

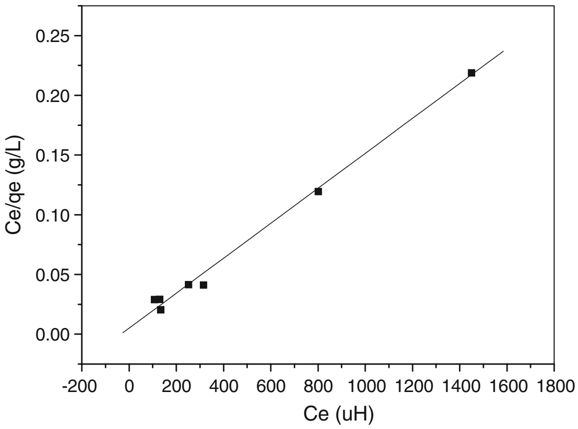

Fig. 5 Linearized Langmuir isotherm plot for biosorption of DIW components (yielding DIW color) by MO biomass. (■) Experimental data; (—) fitting of model

submitted to any chemical pretreatment guarantees that their components, such as proteins and cellulosic compounds, are not altered and can act as ligands in the adsorption process.

The results obtained show that MO seed keeps its adsorption power under a range of pH between 5 and 8. As the DIW presents pH around 7.2, there is no need to adjust the pH for the adsorption process to be efficient. The elimination of the pH adjusting step for the adsorption process in large scale is favorable, simplifying and reducing the process costs.

3.2 Effect of Agitation Time

One of the important physico-chemical aspects for the evaluation of the adsorption process as a unit operation is the sorption equilibrium.

Adsorption data enlightening the effect of agitation time for the partitioning of DIW components between liquid and solid phases is presented in Fig. 2. Agitation time was varied from 5 to 60 min for a 0.2 g sorbent dose and 0.2 L of 1 g/L sorbate solution at pH 7.2 and 25°C. The removal efficiency increased with the increase in agitation time. The equilibrium was accomplished within 60 min. Therefore, for the rest of the study, a 60-min contact time was employed. No further increase in sorption was observed with further increase of contact time up to 40 min. This equilibrium time was reached by Akhtar et al. (2007a) for the removal of organic pollutants using MO pods and by Kumari et al. (2006) for the removal and recovery of arsenic from aqueous system using MO seed powder.

With an agitation time of 60 min, the reduction of both color and turbidity parameters reached 99%. The initial values of wastewater turbidity and color were 912 NTU and 3,515 μH, respectively. After the final step, those values decreased to 1.96 NTU and 25 μH.

Akhtar et al. (2007b) used 0.2 g of chemically treated MO pods and 100 mL of water contaminated with 10 μg mL^{-1} of methyl parathion pesticide, with 90 min of agitation and pH 6. The authors achieved 98% of pesticide removal.

3.3 Effect of MO Amount

In order to investigate the effect of sorbent amount (0.15–0.25 g) on the adsorption of DIW components onto MO using 0.2 L of 1.0 g/L sorbate solution, presented in Fig. 3, a 60-min agitation time was employed at pH 7.2 and 25°C.

A steep increase in removal efficiency was observed from 0.15 to 0.2 g of sorbent amount for turbidity and color parameters, which remained almost constant from 0.2 to 0.25 g for turbidity removal (99.5%).

The COD removal efficiency remained almost constant around 55% for the whole range of sorbent amount.

The extraction of the active coagulant component from *M. oleifera* seed has been developed for water and wastewater treatment by many authors (Okuda et al. 1999; Bhatia et al. 2007; Ghebremichael et al. 2005; Okuda et al. 2001). Reported turbidity removal by coagulant MO was up to 80–99%, but in all cases

Table 2 Langmuir isotherm parameters for DIW uptake by MO biomass

DIW components	$q_{\max}$	K_L	R^2
Turbidity	1,261.45 (NTU × L/g)	0.2037 (1/NTU)	0.9996
Color	6,863.32 (μH × L/g)	0.0294 (1/μH)	0.9966

Table 3 Freundlich isotherm parameters for DIW uptake by MO biomass

DIW components	n	Kf (L/g)	R^2
Turbidity	2.32	292.04	0.8741
Color	2.96	850.20	0.9266

the initial turbidity of the synthetic water was lower than 300 NTU. Considering various aspects like DIW concentration, volume of test solution, and biomass dosage, the present adsorption process is as efficient as the coagulation process using MO extracts. The process using MO as adsorbent has the advantage of a high efficiency for the treatment of wastewater with high levels of turbidity and color.

Another advantage when using MO as adsorbent is the operational simplicity when compared to the coagulation process.

3.4 Adsorption Isotherms

In order to understand the adsorption processes, the Langmuir isotherm (Fig. 4 for turbidity and Fig. 5 for color) was used to represent the equilibrium relationships for experiments with different initial DIW concentrations.

The Langmuir model (Langmuir 1918) represents monolayer adsorption on a set of distinct localized adsorption sites having the same sorption energies independent of surface coverage with no interaction and no steric hindrance between adsorbed molecules and incoming molecules. Adsorption data is also subjected to the linearized form of Langmuir Eq. (3), where $q_{\max}$ and K_L are the Langmuir constants.

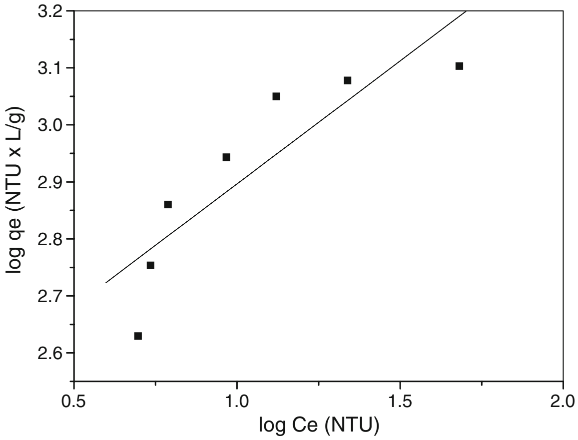

Fig. 6 Linearized Freundlich isotherm plot for biosorption of DIW components (yielding DIW turbidity) by MO biomass. (■) Experimental data; (—) fitting of model

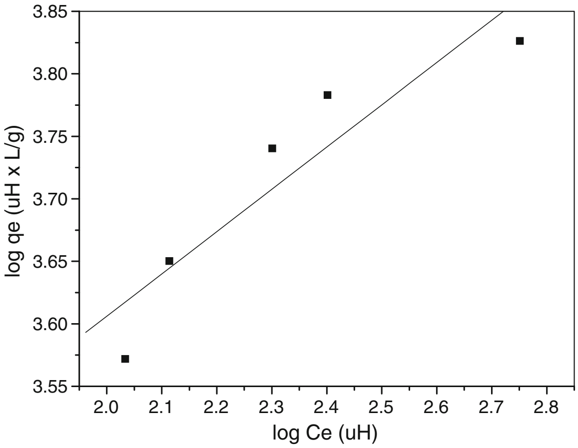

Fig. 7 Linearized Freundlich isotherm plot for biosorption of DIW components (yielding DIW color) by MO biomass. (■) Experimental data; (—) fitting of model

$$\frac{Ce}{qe} = \frac{1}{q_{\max} K_L} + \frac{Ce}{q_{\max}} \tag{3}$$

Freundlich sorption isotherm is the most commonly used empirical expression describing the sorption from solutions and deals with surface heterogeneity, exponential distribution of active sites of sorbent, and their energies towards sorbate (Freundlich 1926) and is given in the linearized form of Eq. (4), where Kf and $1/n$ are constants.

$$\log qe = \frac{1}{n} \log Ce + \log Kf \tag{4}$$

The constants of Langmuir and Freundlich isotherms are tabulated in Tables 2 and 3, respectively. Straight lines were obtained by plotting Ce/qe versus Ce for the DIW components, showing the Langmuir model applicability to sorption data (Figs. 4 and 5).

The values of $q_{\max}$ and K_L estimated respectively from the slope and intercept of the plots are listed in Table 2. The magnitude of $q_{\max}$, the saturation capacity, for turbidity components, indicates that MO biomass can adsorb up to 1,261 NTU of turbidity onto 1.0 g MO/L of DIW solution (1 g/L). As for the color, $q_{\max}$ indicates that MO biomass can adsorb up to 6,863 μH of color onto 1.0 g MO/L of DIW solution (1 g/L).

As indicated in Table 2, the coefficients of determination (R^2) of both parameters, turbidity and color, were greater than 0.99 and close to one, indicating that both parameters adequately describe the experimental data from the biosorption experiments by the Langmuir model.

The plots of log*qe* versus log*Ce* for DIW components, for the application of Freundlich model to sorption data, are presented in Figs. 6 and 7. The obtained parameters are listed in Table 3.

From Table 3, it is apparent that the value of the Freundlich constant *n*, obtained for the present systems, indicates favorable adsorption, as it lies between 1 and 10 (Parab et al. 2006). This indicates effectiveness of MO biosorbent for the removal of DIW components from aqueous solutions. However, the linearized equation did not give a good correlation for both turbidity and color removal from DIW solution onto MO biomass, indicating that the DIW adsorption by MO biosorbent fits better to the Langmuir model than to the Freundlich model.

4 Conclusions

The following conclusions can be drawn from this ongoing study:

- From experimental results, it was concluded that maximum adsorption of DIW components from aqueous solutions occurred using MO biomass doses of 1 g/L with a 60-min agitation time;
- The results obtained show that the MO seed keeps its sorption power under an initial pH ranging from 5 to 8;
- Langmuir model fitted well to the sorption data with higher values of coefficients of determination than Freundlich model.
- The use of MO as a biosorbent is a new method for the treatment of effluents, especially when related to DIW components. The use of MO biosorbent presents operational simplicity and sorption capacity for the removal of organic components from aqueous solutions.
- *M. oleifera* pods are an inexpensive, indigenous, and easily available in large quantities raw material that may be used for different industrial applications to lower the cost of wastewater treatment and it has potential applications for the removal of DIW components.

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