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Green Approach to Wastewater Treatment by Application of *Rosa damascena* Waste as Nano-Biosorbent

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ABSTRACT

Textile wastewater contains many organic and inorganic pollutants and discharging them into received waters leads to serious environmental problems. In this study, rose watering waste (*Rosa damascena*) was used as an abundant and available material for reduction of Chemical Oxygen Demand (COD) and color from textile wastewater in Kashan (Iran). So, rose watering wastes as powder in both bulk and nanometric states were examined and characterized using Scanning Electron Microscope (SEM). The effect of various parameters including contact time (15, 30, 45, 60, 75 and 90 min), pH (3.0, 5.0, 7.0 and 9.0) and nano-biosorbent dosage (500, 1000, 2000 and 4000 mg L⁻¹) on filtered wastewater were investigated. Also, biosorption kinetics for COD was evaluated.

Key words: COD, color, nano-biosorbent, rosa damascena waste, textile wastewater

INTRODUCTION

Textile industries are the most important water pollution resources. Huge amounts of water are used for dyeing in textile mills. About 100 L of water are needed for preparing 1 kg of dyed fabrics (Abadulla *et al.*, 2000; Robinson *et al.*, 2002; Selvam *et al.*, 2003). According to scientific literature, textile wastewater generally contains Biochemical Oxygen Demand (BOD), chemical Oxygen Demand (COD), colors, turbidity, suspended solid, oil and grease, anions and heavy metals such as chloride, sulfide, sulfite, iron, nitrite, nitrate, copper and cadmium (Ghorbani and Eisazadeh, 2013; Ahmad and Hameed, 2009). The COD is a major indicator for assessing organic matter content of wastewater. High COD in a wastewater indicates high level of organic material. Synthetic dyeing agents are organic pollutants determined by measuring the amount of COD (Lv *et al.*, 2009; Thiyagarajan *et al.*, 2013). Some of the dyes from textile industries are nonbiodegradable, toxic, allergen, carcinogen and mutagen for aquatic systems, soil ecosystems and human health (Thiyagarajan *et al.*, 2013; Ahmed *et al.*, 2012; El-Mekkawi and Galal, 2013). Also dyes prevent sunlight penetration and in this way reduce photosynthesis reactions in aqueous environments. Furthermore, dyes impose biological and chemical changes and oxygen deficiency (Wang *et al.*, 2005; Rangabhashiyam *et al.*, 2013; Rodriguez *et al.*, 2009). Consequently, color removal from wastewater is very important and numerous methods such as electrochemical

treatment, chemical oxidation, coagulation, flocculation, reverse osmoses, ultra filtration, chemical precipitation and adsorption has been accomplished for textile wastewater treatment (Thiyagarajan *et al.*, 2013; Rangabhashiyam *et al.*, 2013; Sanroman *et al.*, 2004; Soon and Hameed, 2011; Zahrim *et al.*, 2011; Iscen *et al.*, 2007; Srinivasan and Viraraghavan, 2010). Adsorption is a progressive method because of its ability to remove different kinds of dyeing agents and production of high quality drinking water. Also, recently it has received more attention due to its high efficiency and facility in water and wastewater treatment (Ip *et al.*, 2009; Han *et al.*, 2008). Recently, biosorption as a physicochemical process has been practiced for color removal through inactive biomasses in numerous studies (Thiyagarajan *et al.*, 2013; Guler and Sarioglu, 2013). In biosorption, active sites are generated by biological substances that react with dye molecules (Thiyagarajan *et al.*, 2013). Different kinds of biosorbents have been used for color and COD removal from wastewater such as shrimp shell biomass (Kousha *et al.*, 2015), pine cone (Mahmoudi *et al.*, 2011), tamarindus (El-Mekkawi and Galal, 2013), untreated coffee residues (Kyzas *et al.*, 2012), peanut hull (Tanyildizi, 2011), indica hull (Khorramfar *et al.*, 2009), bamboo-based activated carbon (Ahmad and Hameed, 2009), tree fern (Ho *et al.*, 2005) and glass fibers (Chakrabarti and Dutta, 2005). Many studies have been concentrated on new absorbents with regeneration capability and comfort application in industrial amount. Today nanotechnology products have received more attentions because the technology can change substances to atomic and molecular scales which are more effective than their common sizes. The nanoparticles diameters are in range of 1-100 nm and their specific surface is high (Moussavi and Mahmoudi, 2009; Zhao *et al.*, 2011; Banerjee and Chen, 2007; Liao and Chen, 2002). Nanoparticles have been applied in color and COD removal from industrial wastewaters. Moussavi and Mahmoudi (2009), for example, applied MgO nanoparticles to 50-300 mg L⁻¹ azo and anthraquinone reactive dyes. They stated that in optimum conditions (the optimum dosage of nanoparticles, 2000 mg L⁻¹, pH = 8 and contact time, 5 min) removal efficiency is 98% (Moussavi and Mahmoudi, 2009). Hashemian and Salimi (2012) applied CuFe₂O₄/sawdust nano magnetic composite to dye removal and achieved 90% reduction after contact time of 15 min (Hashemian and Salimi, 2012). Also, Ahmed *et al.* (2012) could remove 97% of color from aqueous solution by 0.1 g of nano-polyaniline and Baker's yeast after 60 min contact time. *Rosa damascena* belongs to the Rosaceae family and is cultivated around the world for its watering and oil extraction. A huge amount of rose flower is needed for production of a little rose water (Tsanaktsidis *et al.*, 2012; Nasir *et al.*, 2007). So, the process generates large amount of semisolid wastes that can lead to environment pollution.

A traditional livelihood in Kashan (Iran) rural area was rose watering which has been industrialized today. Now-a-days Qamsar rose water is very popular in the world especially in Asia. Furthermore, textile wastewater is one of the environmental problems in Kashan because of textile industry development. Accordingly, in this study a nano-biosorbent was produced from dry rose watering wastes (*Rosa damascena*) and its application was investigated to textile wastewater treatment.

MATERIALS AND METHODS

Materials: Aerial parts of *Rosa damascena* were collected during June, 2013 from Kashan area (Isfahan Province, Iran) at an altitude of ca. 1100 m. After completion of rose watering procedures, wastes were collected and used as biosorbent in the study.

Preparation of the biosorbent: First, a sufficient mass of the waste was rinsed by distilled water for dust removal. Next the rinsed waste dried in a furnace at a temperature of 70°C for 48 h. The waste was submerged in 0.2 M H₂SO₄ solution for 60 min and rinsed again. Then, it was soaked in 0.2 M NaOH solution for 1 h and rinsed by distilled water. Next, it was placed in an oven at 550°C for 1 h for removal of VOCs. Subsequently, rose watering wastes as powder in both bulk and nanometric states were prepared.

In order to obtain nano-biosorbent, the residue was grinded and milled in a planetary high energy ball mill using hardened chromium steel vial and balls. Ball milling was performed at room temperature. The rotational speed was 600 rpm. Sieved residue was stored in plastic containers. The size of particles was determined by a LEO1455VP model Scanning Electron Microscope (SEM).

Biosorption activity: A raw wastewater sample was taken from effluent of equalization tank of industrial wastewater treatment plant in a textile mill in Kashan (Iran). The color of sample was dark brown. Chemical Oxygen Demand (COD), color intensity and pH were measured in accordance with primary goal for removal of COD and color from wastewater sample and these were determined 1840 mg L⁻¹, 70% and 10.5, respectively. The sample was kept below 4°C for prevention of changes in the wastewater characteristics (Rice *et al.*, 2012). First, by a predetermined dosage of the nano-biosorbent at constant pH, the optimum contact time was determined. Then, the experiments were followed by the results obtained in this stage. After that, in each run a certain dosage (500, 1000, 2000 and 4000 mg) of the prepared nano-biosorbent was added to 1.0 L of raw wastewater sample in a batch reactor and one sample was taken at 45 min contact time from the batch content for COD and color analysis. These processes were repeated for the initial pH of 3.0, 5.0 and 9.0. The pH was adjusted by 0.2 M solution of H₂SO₄/NaOH with pH meter (Iran; Fanavary Taghizat Sangesh, pH 262). Finally, according to the obtained results in the previous stages, biosorption kinetic of COD was studied in contact time of 15-90 min.

All experiments were accomplished at laboratory temperature (about 25°C). Samples shaking were done in 240 rpm by an HMS8805 device (Iran; Pol Ideal, HMS8805). After 10 min of settling nanoparticles, samples were filtered and prepared for experiments. The COD was analyzed by 5220-C method. Color intensity was determined in 30 wavelengths by 2120-C method using (USA; APEL, PD-303UV) spectrophotometer (APHA, AWWA and WPCF., 2005). Then color removal percent was calculated based on color intensity.

Statistical analyzes: The experimental results were analyzed by spss 16 software for ANOVA test to determine significant difference between the effect of various pH and biosorbent dose on COD and color removal and Microsoft Excel 2010.

RESULTS AND DISCUSSION

Characteristics of nanoparticles of rose watering waste (*Rosa damascena*): Scanning electron micrographs of nanoparticles is shown in Fig. 1. The average particle size of the absorbent after milling was in the range of 70-80 nm. The effect of this biosorbent on the textile wastewater characteristics was investigated for both bulk and nano-biosorbent.

Effect of contact time: The maximum efficiency of COD and color removal obtained after contact time of 45 min were 59.78 and 45.71%, respectively (Fig. 2). Figure 2 shows the role of contact time in the range of 15-90 min in COD and color removal.

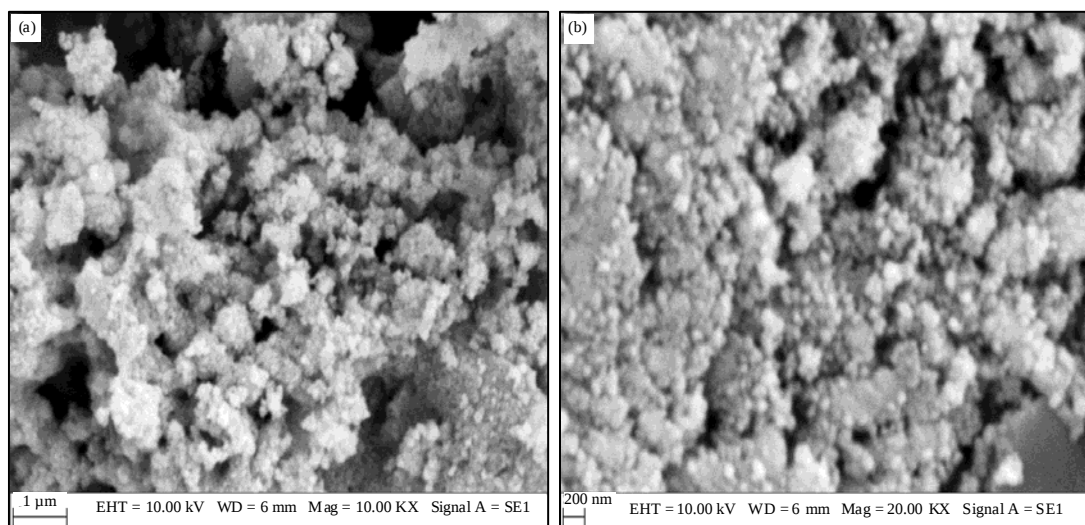


Fig. 1(a-b): Scanning electron microscopy micrographs of the prepared nanoparticles of rose watering waste (*Rosa damascena*)

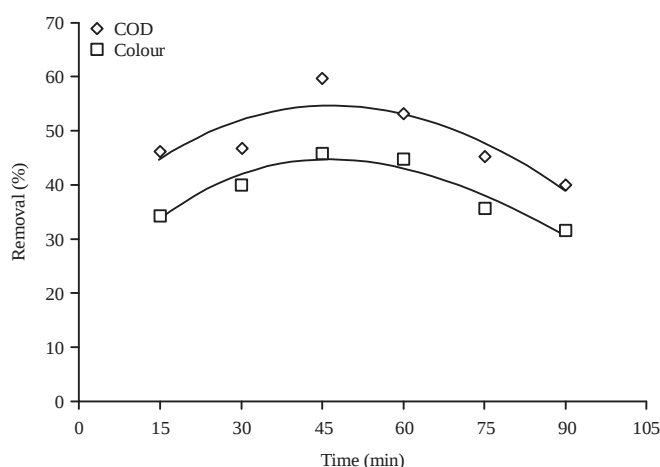


Fig. 2: Effect of contact time on COD and color removal in optimized pH and nano-biosorbent dosage of rose watering waste (*Rosa damascena*), COD: Chemical oxygen demand

Contact time is an effective key factor on adsorption process efficacy (Moussavi and Mahmoudi, 2009). The findings showed that the rate of adsorption in the first 15 min of contact time was more than others which can be related to the existence of numerous available active functional groups in the beginning (El Boujaady *et al.*, 2014). After 15 up to 45 min, the biosorption was increased but with lower rate. At contact time longer than 45, the removal of COD and color were decreased which can be related to desorption of the dye molecules from the nano-biosorbent surface due to repellent forces between dye molecules at adjacent sites on the nano-biosorbent surfaces.

Srivastava *et al.* (2005) reported equilibrium time of 60 min for removal COD and color from pulp and paper mill wastewater with bagasse fly ash.

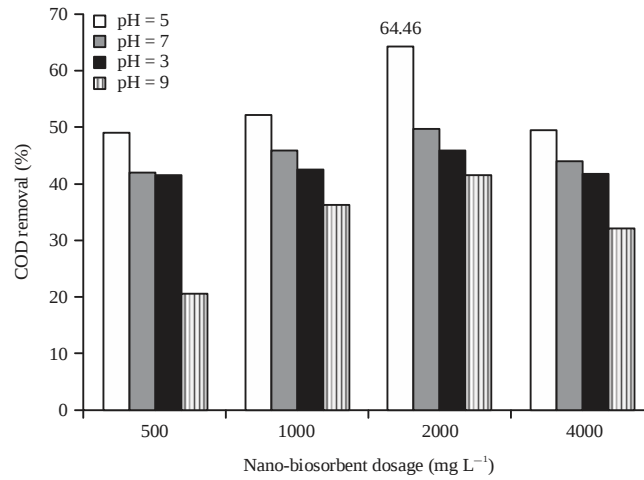


Fig. 3: Effect of pH in different biosorbent dosages of the nano-biosorbent on COD removal, COD: Chemical oxygen demand

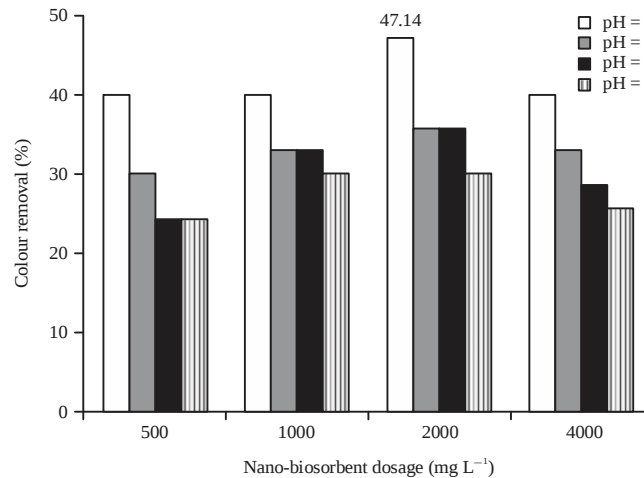


Fig. 4: Effect of pH in different dosages of the nano-biosorbent on color removal

Effect of pH and nano-biosorbent dosages: The effect of pH on COD removal in different dosages of the nano-biosorbent of rose watering waste is shown in Fig. 3. It can be seen that pH = 5.0 and 2000 mg L⁻¹ of the nano-biosorbent are regarded as the best conditions in the study.

Also the effect of pH in different dosages of the nano-biosorbent on color removal from textile wastewater is shown in Fig. 4. The optimum conditions are pH = 5.0 and nano-biosorbent dosage of 2000 mg L⁻¹. Comparing Fig. 3 and 4 shows the optimum conditions of COD and color removal by this process was the same. The nano-biosorbent of rose watering waste was more efficient for COD removal than color. According to the ANOVA analysis, the effect of pH and dosage of nano-biosorbent on COD removal was more significant than color ($p < 0.05$). The pH of solution affects adsorption process of dye molecules by influencing aqueous chemistry and binding sites. So, it seems that this parameter plays an important role in controlling the adsorption process (Nasir *et al.*, 2007; Madrakian *et al.*, 2012). The decreasing in the removal at very acidic

pH (pH = 3.0) is due to the surface of nano-biosorbent is surrounded by the excess hydronium ions that compete with cationic dye for active sites on nano-biosorbent (Zhou *et al.*, 2011). At alkaline pH, the presence of excess hydroxyl ions competing with anionic dyes for nano-biosorption sites resulted decrease in removal (Ncibi *et al.*, 2009). Madrakian *et al.* (2012) observed similar trend for removal neutral red dye by magnetite nanoparticles loaded tea waste and reported maximum adsorption of neutral red at pH 5-6. Aksu (2001) reported maximum removal efficiency of reactive yellow by dried activated sludge at pH = 5. Srivastava *et al.* (2005) observed maximum COD and color removal in the treatment of pulp and paper mill wastewater by bagasse fly ash at pH = 4.0.

Figure 3 and 4 show that increasing nano-biosorbent dosage to 2000 mg L⁻¹ increases removal rate of pollutants. This performance is due to the increase in the usable nano-biosorption sites with increasing in nano-biosorbent dosage (Hameed, 2009). Reduction in the removal rate at nano-biosorbent dosage of 4000 mg L⁻¹ is that with increasing dosage intervals between nano-biosorbent particles reduce and the binding sites are not fully occupied (Ahmed *et al.*, 2012). Furthermore, at high nano-biosorbent dosage, the interaction between particles such as overlapping and aggregation occurs and the number of available surface area reduces (Isken *et al.*, 2007). Generally the biosorption process with nanoparticles of rose watering waste due to electrostatic attractive forces between adsorbent and absorbent (Alley, 2000). Srivastava *et al.* (2005) reported optimum dosage of 2 g L⁻¹ of bagasse fly ash for the removal of COD and color from pulp and paper mill wastewater treatment. Moussavi and Mahmoudi (2009) observed the maximum removal of azo and anthraquinone reactive dyes from industrial wastewaters under optimum dosage of MgO nanoparticles of 0.2 g/100 mL.

Biosorption kinetics: To evaluate the biosorption kinetics, pseudo-first-order and pseudo-second-order kinetics models were selected. The pseudo-first-order equation was presented by Lagergren (1898) as Eq. 1:

$$\log (q_e - q_t) = \log q_e - \frac{k_1 \cdot t}{2.303} \quad (1)$$

And pseudo-second-order equation was presented by Ho and McKay (1999) as Eq. 2:

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (2)$$

where, q_e (mg g⁻¹) is adsorption capacity at equilibrium time, q_t (mg g⁻¹) is adsorption capacity at time t (min) and K_1 (min⁻¹) and K_2 (g mg⁻¹ min⁻¹) are rate constants.

Kinetics biosorption of COD by the nanoparticles of rose watering waste were evaluated at contact time of 15-90 min, pH = 5 and nano-biosorbent dosage 2000 mg L⁻¹.

Figure 5 shows the plot of t/q_t vs. t for the pseudo-second-order kinetic model. Values of q_e and K_2 were calculated from the slope and intercept of Fig. 5 and summarized in Table 1. The plot of pseud-first-order model is not shown because the correlation coefficient of the pseud-first-order model was lower than that in the pseudo-second order model. Therefore, the biosorption of COD follows the pseudo-second-order kinetic model ($R^2 = 0.9504$).

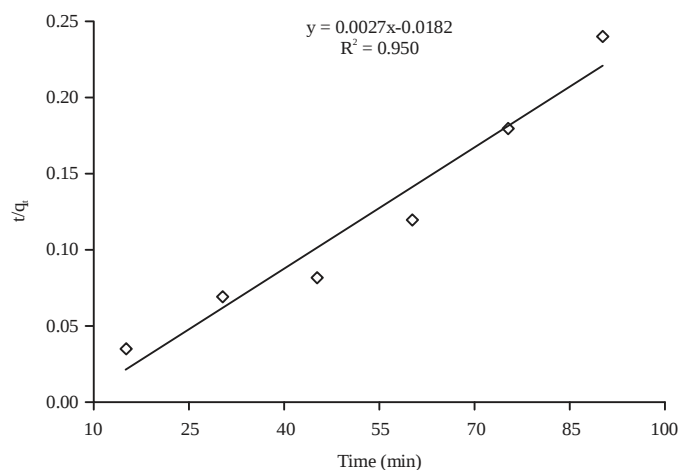


Fig. 5: Pseudo-second-order kinetic plot for biosorption of COD by nano-biosorbent

Table 1: Pseudo-second-order kinetic parameters for biosorption of COD by nanoparticles of rose watering waste (*Rosa damascena*)

Parameters	Values
R^2	0.9504
q_{exp} (mg g ⁻¹)	550
q_e (mg g ⁻¹)	370.37
K_2 (g mg ⁻¹ min ⁻¹)	0.0004

COD: Chemical oxygen demand

An important stage for designing biosorption system is determining the biosorption kinetics. The information of the biosorption kinetics is required to determine the optimum conditions for full scale batch systems (Akar *et al.*, 2009).

The COD biosorption was better fitted by pseudo-second-order kinetic model. Following COD biosorption from this kinetic model has been reported by Srivastava *et al.* (2005) for the treatment of pulp and paper mill wastewaters with poly aluminium chloride and bagasse fly ash and El-Naas *et al.* (2010) for the reduction of COD in refinery wastewater through adsorption on date-pit activated carbon.

CONCLUSION

The aim of this study was to evaluate the capacity of nano-biosorbent of rose watering waste (*Rosa damascena*) in textile wastewater treatment. The results from the investigation of the studied parameters showed that the optimum conditions toward maximum removing value of COD and the color were contact time of 60 and 45 min for bulk and nano-biosorbent, respectively, as well as pH = 5.0 and biosorbent dosage of 2000 mg L⁻¹. Also, the capability of rose waste to remove COD was higher than the color. Biosorption kinetic for COD for both biosorbent followed pseudo-second-order model ($R^2 = 0.911$ for bulk and $R^2 = 0.9504$ for nano-biosorbent). According to the results, the capacity of nano-biosorption in treating textile industry wastewater was more than that of bulk biosorption. Generally, increasing surface to volume ratios in nanomaterials enhance their catalytic activity and adsorptivity capacity which is due to increasing in available sites for interactions the species. Processing waste materials (such as rose waste) and converting into useful and efficient materials is an important subject has recently gained attractions of many researchers.

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