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Evaluation of the *Peganum harmala* plant stems in the removal of total dissolved salts from water

S.M. Abdel Azeem[a,b,*], Magdy D. Madbouly[b,c], M.F. El-Shahat[d]

[a]*Chemistry Department, Faculty of Science, Fayoum University, Egypt, Tel. +201207664342, Fax +20846370025*
email: sma13@fayoum.edu.eg (S.M.A. Azeem)
[b]*Chemistry Department, Al-Quwayiyah College of Science and Humanities, Shaqra University, Kingdom of Saudi Arabia*
[c]*Environmental Pollution Department, The National Centre for Social and Criminological Research, Cairo,*
Egypt, Tel. +201120813468, email: magdiab@hotmail.com (M.D. Madbouly)
[d]*Chemistry Department, Faculty of Science, Ain–Shams University, Cairo, Egypt, Tel. +20224196561,*
email: elshahatmf@hotmail.com (M.F. El-Shahat)

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ABSTRACT

A low-cost and eco-friendly solid-phase extraction sorbent obtained from *Peganum harmala* stems was developed. It was used for the removal of total dissolved salts (TDS) from non-drinking tap water derived from boreholes. The prepared sorbent was characterized with FT-IR and SEM micrograph. Adsorption parameters were investigated by the batch method. Results showed a significant decrease in TDS by increasing the shaking time. Adsorption kinetics followed the first-order kinetics with a half-life time of 16.1 min. Adsorption isotherm obeyed Freundlich physisorption model with a correlation coefficient of 0.956. Thermodynamic study proved an endothermic adsorption with a heat of adsorption of 8.1 J/mol. Under dynamic mode, results showed a gradual decrease in TDS and reached values of 840, 735 and 802 mg/L for *Peganum harmala* filter, and commercial sediment and carbon filters, respectively after flowing time of 100 min. Also, passing tap water for three days gave values of 691, 550 and 600 mg /L, respectively. Therefore, the developed *Peganum harmala* filter conveyed a comparable efficiency to the commercial sediment and carbon filters.

Keywords: Peganum harmala; Solid-phase extraction; Total dissolved salts; Tap water

1. Introduction

High levels of total dissolved salts (TDS) in water may be repugnant to consumers due to the undesirable taste and excessive scaling in water pipes, heaters, boilers and household appliances. It is also often corrosive to water-supply systems. Controversy, water with extremely low concentrations of TDS may also be unacceptable because of the insipid taste [1].

The use of plants to clean up soil and water contaminated with heavy metals is a new technology known as phytoremediation [2]. A type of phytoremediation called rhizofiltration which includes the use of plants to remove heavy metals from aqueous streams [3].

Certain plants can concentrate the essential and non-essential heavy metals in their roots and shoots to levels higher than those present in the soil. These plant species are restricted to metalliferous soils. The mechanisms of metal accumulation involve extracellular and intracellular metal chelation, precipitation, compartmentalization, and translocation in the vascular system [4].

Peganum harmala (*P. harmala*) or esphand is a perennial plant. It belongs to the family Nitrariaceae, native from the eastern Mediterranean region east to India [5]. It is also known as Wild Rue or Syrian Rue because of its resemblance to plants of the rue family which is commonly used for folk medicine in many countries. Various parts of the plant are used in folk medicine to treat several illnesses. The plant is drenched in water and the infusion liquid is usually orally taken [6]. The seeds were used in traditional

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*Corresponding author.

Moroccan medicine in the form of powder or beverage for the treatment of fever, diarrhea subcutaneous tumors, treatment of rheumatic and gastrointestinal aches, asthma, jaundice, low back pain and for abortion. Further, it haspharmacological action against viruses, cancer, convulsions and fever besides its use as a hallucinogen, antibacterial and antiprotozoal [7]. In Iran and Turkey, they burn the seeds to remove the evil spirits [8].

The active compounds in *P. harmala* belongs to b-carboline alkaloids which are a group of alkaloids called haraman alkaloids. These alkaloids are mainly located in the leaves, roots, stems and seeds of *P. harmala*. The identified b-carboline alkaloids in *P. harmala* extracts are harmaline, harmine, harmalol, harmol and tetrahydroharmine. The level of these alkaloids is higher in seeds and roots than in the stems and leaves [9]. Harmine and harmaline accumulated in dry seeds at 4.3% and 5.6% (w/w), respectively, harmalol at 0.6%, and tetrahydroharmine at 0.1% (w/w). Roots contained harmine and harmol with 2.0% and 1.4% (w/w), respectively [9,10].

Removal of toxic metal ions by *P. harmala* was reported in literature. A biosorbent from *P. harmala* seeds for adsorption of lead, zinc and cadmium [11] cadmium [12], and nickel [13] from contaminated water was investigated. Green synthesis of zinc oxide nanoparticles using *P. harmala* seed extract loaded on the plant seed and powdered activated carbon was used for removal of Cr(VI) from aqueous solution [14]. The seed extract was examined to protect iron alloy from corrosion [15]. Removal of the brilliant green [16] and methylene blue [17] dyes were reported. A continuous flow adsorption method was published for quantification of heavy metal ions by *P. harmala* seeds [18].

The TDS refer to any minerals, salts, metals, cations or anions dissolved in water due to natural sources, sewage, urban run-off, industrial wastewater, and chemicals added during the water treatment [19]. These dissolved species comprise inorganic ions such as calcium, magnesium, potassium, sodium, bicarbonates, chlorides and sulfates, as well as some small amounts of soluble organic matter [20].

The Environmental Protection Agency (EPA) classified the TDS as a secondary contaminant and suggested a maximum limit of 500 ppm. The value of TDS represents the sum of the cations and anions ions in water. Therefore, it is considered as a qualitative test to measure the amount of dissolved ions. On the other hand, it does not tell us about the nature of ion relationships, the specific water quality issues, elevated hardness, salty taste, or corrosiveness [21].

The hazardous of TDS can be seen in the murky appearance of water, and they can detract from water's taste and quality. High TDS level can cause gastrointestinal irritation in some individuals [22]. Moreover, the TDS can interfere with treatment devices, thereby, they should be considered when choosing a treatment system.

Determination of the TDS can be carried out by gravimetric analysis [23] and conductimeteric [24] analysis. Gravimetric analysis requires filtering of a measured volume of water sample through a sintered glass fiber. The filtrate is then dried at 103°C in a pre-weighed ceramic dish then heated at 180°C. The weight of remaining residue represents the TDS according to the method specified by Code of Federal Regulations (40 CFR) Part 136 [25]. Electric conductivity (EC) measurement is an indirect quantification of TDS in water. A higher value means water is a better electrical conductor and consequently high concentration of dissolved salts. The TDS (mg/L) is estimated by multiplying EC with an empirical factor varying from 0.55 to 0.90, depending on the type of soluble components and the solution temperature [26].

The aim of the present work is to use the widely distributed perennial plant at Al Quwayiyah governorate called *P. harmala* for the removal of TDS from non-drinking tap water derived from boreholes. The quality parameters for two boreholes waters and drinking bottled water samples were measured. The adsorption kinetics, isotherm, and thermodynamics were investigated via the batch technique. Finally, a continuous flow method was set up using a developed filter packed with the sorbent under study and compared to the commercial sediment and carbon filters.

2. Experimental

2.1. Instruments and reagents

A laboratory spectrophotometer model DR3900 obtained from Hach company (Colorado, USA) was used to measure several species in water, such as Fe, Mn, sulfate, nitrate, nitrite, fluoride, and free chlorine. A pH and turbidity meters were utilized to monitor the quality parameters of water samples. Jenway bench electrical conductivity (EC) meter model 4510 (Keison International Ltd., Chelmsford, UK) was used for monitoring the EC, TDS and temperature. The TDS range and accuracy are 0–1999 g/L and ±0.5% ±2, respectively. Commercial sediment filter was purchased from a local market and used for the fabrication of the developed filter. Polyethylene tubes with 50 mm i.d were used for all connections. Tap water was connected to the *P. harmala* packed filter and the TDS was measured under dynamic mode.

2.2. Preparation of P. harmala sorbent

The *P. harmala* is a local and widely distributed desert plant in Al Quwayiyah governorate. Bedouins usually add it to borehole water for treatment of sheep and camel against diseases. The choice of the *P. harmala* stems was based on their low content of harmine alkaloids that can chelate to inorganic ions in water. The stems contains less harmine alkaloids content than those in the seeds or roots [9]. On the contrary, high amount of alkaloids in the sorbent is not recommended since these compounds maybe leached into the treated water. Fresh stems of *P. harmala* plant were obtained from a nearby desert area at Al Quwayiyah governorate. They were naturally dried and ground into fine particles in a porcelain mortar. The powdered material was sieved to a particle size lower than 0.2 mm. Then, it was washed successively with 1.0 mol/L hydrochloric acid, distilled water and finally with acetone then left to dry in ambient atmosphere. The purpose of washing with acid and acetone is to remove inorganic matrices and organic pigments in the sorbent. Finally, the powdered material was washed with distilled water till the effluent was free from any leached pigments. The possibility of leaching out of the pigments into the treated water was examined. For this purpose, a 0.5 g sorbent was shaken in 40 mL distilled water for 1 h. After that, the sorbent was separated and the absorbance was measured over a wavelength range from 200 to 800 nm. No absorbance signal was detected. This confirmed no organic matters were added to the aqueous solution.

2.3. Water sampling

Tap, non-drinking, water samples were collected from our laboratory at chemistry department. Borehole waters 1 and 2 were collected from two different locations in Al Quwayiyah governorate. All samples were placed in plastic bottles then analyzed without any further treatment. Adsorption and thermodynamic studies were operated on the same day of sample collection. The synthetic solutions were made by diluting tap water obtained from boreholes using distilled water. The composition of tap water is not fixed as it is weekly fed to the institution by local suppliers from various sources. Thereby, the TDS of tap water was measured before and after dilution prior to the adsorption experiments.

2.4. Preparation for TDS measurement

The EC is directly proportional to the concentration of dissolved ionized solids in water. These dissolved ions enable conducting an electric current. Value of EC provides an approximate concentration of TDS with an accuracywithin 10%. The relation between TDS (mg/L) and EC (µSiemens/cm) is given by Eq. (1):

$$TDS = EC * 0.64 \tag{1}$$

Synthetic water samples were prepared at varying TDS levels from 12.5 to 75 mg/L by a serial dilution of tap water (1030 mg/L) with distilled water. After adsorption, the sample was filtered through Whatman filter paper and the EC was measured. The conductivity meter affords to toggle between the EC and TDS results. Operating conditions were optimized according to the manufacturer [27]. To get high accuracy, no suspended particulates or trapped air bubbles were allowed to exist between the electrode plates in the cell area. The complete dipping of the conductivity probe in the solution and the reference temperature were always checked. After use, the cell plates were rinsed thoroughly with distilled water to prevent the formation of salt deposits which may produce negative errors in the subsequent conductivity measurements.

2.5. Recommended procedures

2.5.1. Static removal of TDS

An aliquot 40 mL of tap water was accurately transferred into a 100 mL glass beaker and a 0.25 g sorbent was added. The shaking time was changed from 5 to 100 min at room temperature. Then, the sorbent was filtered off through Whatman filter paper No. 1 and the remaining dissolved salts were measured as TDS (mg/L) using the conductivity meter. Adsorption isotherm was studied using 40 mL samples containing different concentrations of TDS at 12.5, 25, 37.5, 50, 62.5 and 75 mg/L. To each sample, a fixed amount of a 0.25 g adsorbent was added then it was shaken for 1.0 h at ambient temperature and the corresponding TDS were evaluated. Finally, the influence of temperature on adsorption was tested at 20, 30, 50, and 70°C using 40 mL samples containing TDS level of 1030 mg/L, 0.25 g sorbent and shaking time of 1.0 h.

2.5.2. Dynamic removal of TDS

The setup for the dynamic removal of TDS is presented in Fig. 1. A homemade *P. harmala* filter used for removal of TDS was fabricated. For this purpose, a commercial sediment filter was packed with 20 g of the sorbent in the area between the inner wall of the filter and the central tube. Tap water supply and treated water outlet were connected to the filter using polyethylene tubes of 50 mm i.d. Under these conditions, water passes through the sorbent into the hollow central sediment filter made of polypropylene fibers. Then, the treated water leaves the filter towards the measuring EC electrode. The tube carrying the water outlet was fixed into the EC electrode. The emerged effluent passes to the drain via two side holes in the glass part of the electrode. Water stream was kept flowing at a constant rate of 100 mL/min between the two metallic plates in the electrode and the EC was recorded continuously against time.

3. Results and discussions

3.1. Characterization of the sorbent

The FT-IR spectrum of the prepared sorbent from the *P. harmala* stems is presented in Fig. 2. Characteristic broad absorption band appeared for OH and NH at 3414.0 cm^{-1}, for =CH stretching at 2918.9 cm^{-1}, for C=N at 1634.7 cm^{-1}, for C-O at 1464.8 cm^{-1}, and for C=N bending at 1267.9 cm^{-1}. The band for OH group revealed the existence of the harmalol compound. All these peaks confirmed the active functional groups of the β-carboline alkaloids in *P. harmala* [28].

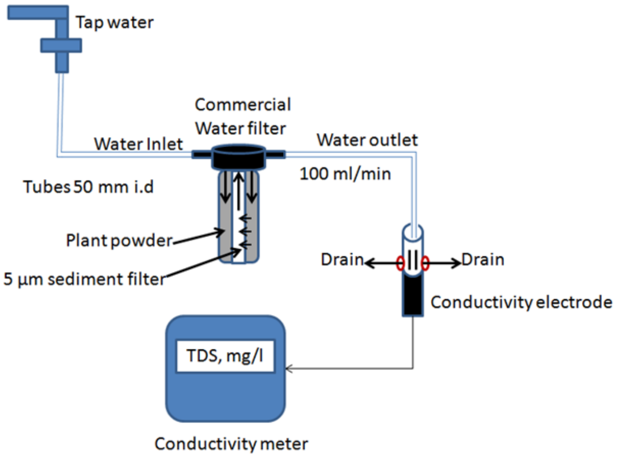

Fig. 1. A schematic diagram of the setup used for dynamic removal of TDS from water.

The SEM micrograph of *P. harmala* at different scales is shown in Fig. 3. Obviously, the sorbent surface appeared as highly porous, irregular, deep, and heterogeneous which will assist the occurance of an extensive adsorption of dissolved ions in water.

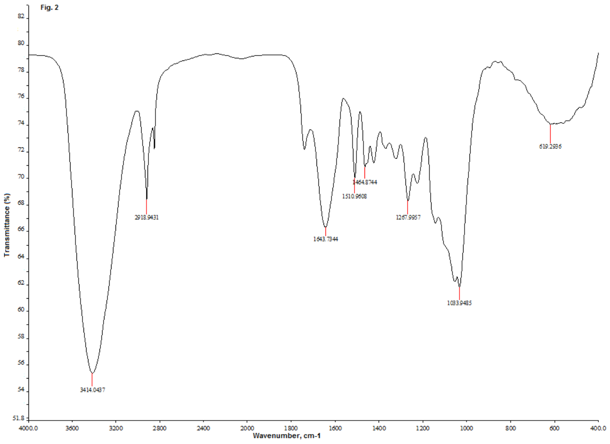

Fig. 2. FT-IR spectrum of P. *harmala* adsorbent.

3.2. TDS and water quality

The main dissolved ions in water are calcium, chloride, nitrate, phosphate, iron, sulfur, and other ions that will pass through a filter with pores of around 2 µm sizes. Besides, suspended solids which may exist in water include silt and clay particles, plankton, algae, fine organic debris, and other particulate matters, but these particles cannot pass through a 2 µm filter. The cloudiness of water can alter the transmitted light path due to hitting the particles in water [29]. Also, the dissolved colored and fluorescent matters and organic dyes contribute to water turbidity [30].

The water balance in the cells of aquatic organisms is affected by the value of TDS. At a very low TDS level, water tends to move into the cells that contain higher concentration of solids and the organism will swell up. Controversy, the organism that lives in water of a high concentration of TDS will shrink because water tends to move out its cells. Accordingly, the TDS level strongly influences the organism's ability to maintain the proper cell density. In addition, higher levels of TDS can also clog the irrigation devices and might become so high that the irrigated plant roots will lose water rather than gain it. Furthermore, elevated concentrations of suspended solids can serve as carriers of toxins such as pesticides to the extent that when water trav-

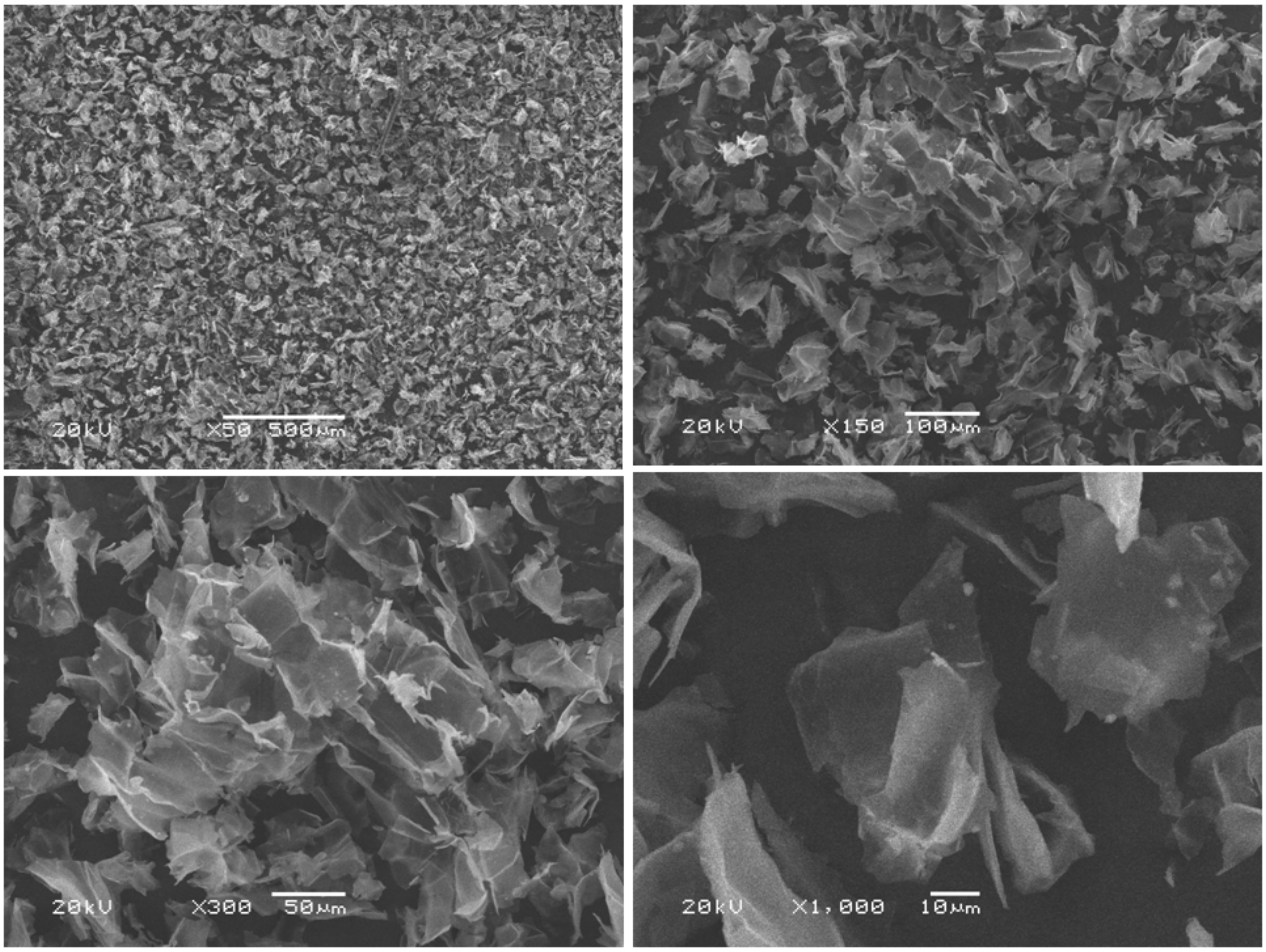

Fig. 3. Scanning electron micrograph of *P. harmala* adsorbent at different scales.

els down irrigation ditches, the concentration of pesticides might exceed those of the original application [31].

3.3. Analysis of water samples

In order to investigate the quality of water in the studied region, the borehole water samples were taken and analyzed then compared to bottled drinking water bought from the local market. The obtained results are presented in Table 1.The pH measurements indicated values of 7.2, 7.2, and 6.9 for borehole 1, borehole 2, and bottled water, respectively which are in a good agreement with the optimal value of fresh water. This indicates an adequate acidity of the analyzed water samples. Turbidity values were found in the order borehole 2 > borehole 1 > bottled water. The TDS analysis was measured in mg/L for the three samples. Results indicated higher TDS for borehole 2 than borehole 1 followed by drinking water. The TDS for drinking water should not be high, otherwise, it will make it unpalatable and may adversely affect people who are not used to drinking such kind of water.

3.4. Static removal of TDS

3.4.1. Effect of sorbent dose

The influence of sorbent dose on the removal of TDS was examined within a range of 0.05–0.5 g. An aliquot of tap water (40 mL) containing TDS concentration of 1030 ± 10 mg/L was shaken with the sorbent for one hour. The results obtained are shown in Fig. 4. Obviously, the sorbent amount up to 0.1 g showed TDS value around 1580 mg/L. Beyond 0.2 g and upward, the value of TDS decreased rapidly and reached a value of 430 mg/L at a sorbent weight of 0.5 g. Thus, an adequate amount of sorbent was optimized according to the degree of the required TDS level.

Table 1
Quality parameters for borehole and bottled drinking water samples

Quality parameter	Borehole 1	Borehole 2	Bottled drinking Water
pH	7.2	7.2	6.9
Turbidity, NTU	0.67	0.78	0.29
TDS, mg/L	941.0	1066.0	153.1
Conductivity, µs/cm	1360.0	1527.0	239.1
Iron (Fe), mg/L	0.12	0.9	0.13
Manganese (Mn), mg/L	0.5	0.2	0.3
Sulfate (SO_4^{2-}), mg/ L	119	126	31
Nitrate (NO_3^-), mg/ L	31	10.4	13.8
Fluoride F^-, mg/ L	1.04	0.83	0.25
Nitrite (NO_2^-), mg/ L	0.01	0.014	0.004
Free Chlorine Cl_2, mg/ L	0.02	0.01	0.02

NTU: Nephelometric Turbitity Unit.

3.4.2. Adsorption kinetics

The effect of shaking time on the removal of TDS from tap water was examined in the range 2–20 min. It can be observed from Fig. 5 that a fast increase in the removal of TDS was achieved up to 60 min. Then, the percentage of removal of TDS became smaller and approximately leveled off at around 80 min. Maximum removal (49%) of TDS was achieved at 100 min. This confirmed the effectiveness of the proposed sorbent to get rid of a tremendous amount of TDS within a short time. To specify the kinetic mechanism of adsorption, the results obtained were examined to fit two kinetic models. Firstly, the Lagergren and Kungliga pseudo-first-order kinetic model [32] described by Eq. (2) was examined:

$$\log\left(q_e - q_t\right) = \log q_e - \frac{k_{ad}}{2.303}t \qquad (2)$$

where q_e and q_t are the adsorbed amount of TDS (mg/g) at equilibrium and any time t (min), respectively and K_{ad} is

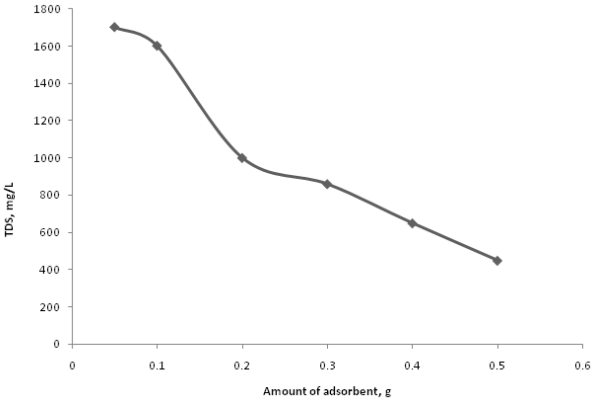

Fig. 4. Effect of sorbent amount on the removal of TDS from tap water.

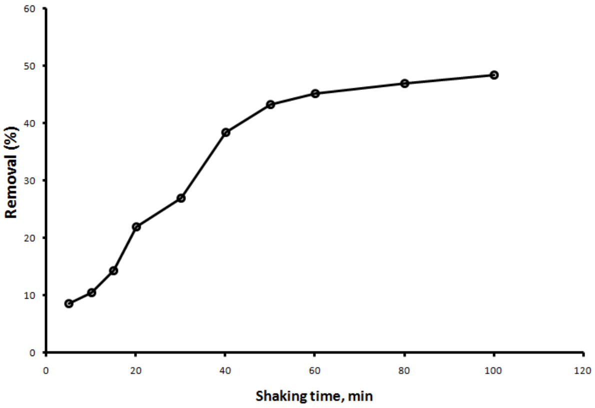

Fig. 5. Effect of shaking time on the removal of TDS from tap water using 0.25 g sorbent and 40 mL water sample at 25°C.

the pseudo-first-order rate constant (min^{-1}). Plotting of log ($q_e - q_t$) versus t is shown in Fig. 6. Results showed a good correlation coefficient of $R^2 = 0.944$.

Secondly, the Ho's pseudo-second-order kinetic model [33] is presented in Eq. (3):

$$\frac{t}{q_t} = \frac{1}{kq_e^2} + \frac{1}{q_e} \tag{3}$$

The results obtained are plotted in Fig. 7. The correlation coefficient R^2 was 0.884 which is lower than that corresponding to the first-order kinetic model. This indicates good applicability of the pseudo-first-order kinetic model to describe the adsorption kinetics of TDS onto the plant adsorbent. Thus, the adsorption process is mainly dependent on the adsorption sites rather than the adsorbed ions. Finally, the rate constant and half-life time were found to be 0.043 min^{-1} and 16.1 min, respectively.

3.4.3. Adsorption isotherm

The effect of TDS concentration was examined within a range of 0.0–1100 mg/L using 40 mL of tap water. The shaking time was fixed at 1.0 h with absorbent weight of 0.25 g. The adsorption capacity of *P. haramala* stems (mg/g) versus the TDS concentration (mg/L) is displayed in Fig. 8. A linear relationship was obtained up to a concentration of 600 mg/L. Beyond this limit, the profile becomes non-linear and the adsorbed amount reached a constant value of 220 ± 4.6 mg/g.

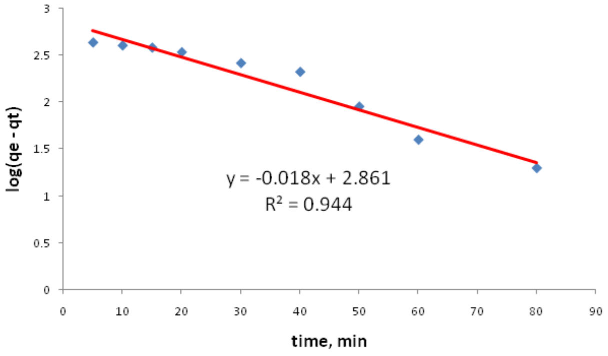

Fig. 6. Lagragren plot for adsorption of TDS in tap water onto the plant adsorbent using 0.25 g sorbent and 40 mL tap water sample at 25°C.

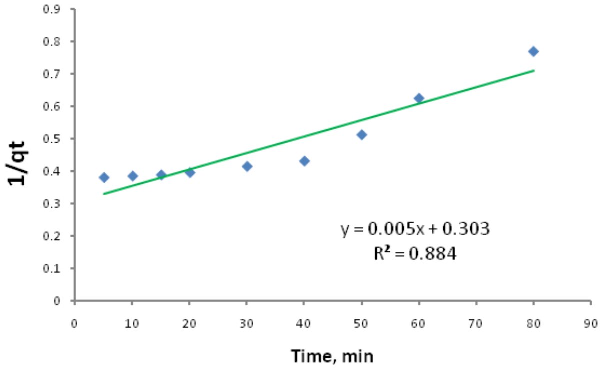

Fig. 7. Second-order kinetic plot for adsorption of TDS in tap water onto the plant adsorbent using 0.25 g sorbent and 40 mL tap water sample at 25°C.

3.4.4. Isotherm models

The experimental results for adsorption isotherm were tested to fit Langmiur and Freundlich models. The Langmuir model [34] is represented by the linearized equation (4):

$$C_e/q_e = 1/K_L q_m + C_e/q_m \tag{4}$$

where C_e is the equilibrium concentration of TDS (mg/L), q_m is the adsorbed amount of salts (mg/g) and K_L is the Langmiur constant (L/mg). This model is the most widely applied as representative of monolayer adsorption of non-interactive ions onto an energetically uniform surface. The plot of Langmiur isotherm model is shown in Fig. 9.

Freundlich model [35] was used to estimate the adsorption intensity of TDS towards the adsorbent surface. It can be interpreted by the linear equation (5).

$$\ln q_e = \ln K_F + (1/n) \ln C_e \tag{5}$$

The plotting of experimental data by Freundlich model is depicted in Fig. 10. Comparing the isotherm parameters obtained from the two models revealed the adsorption was better correlated to the Freundlich model ($R^2 = 0.956$) than Langmuir one ($R^2 = 0.912$). Freundlich constant n was 1.6 which proved a good affinity of TDS to the active sites in the sorbent surface.

3.4.5. Effect of temperature

In order to evaluate the thermodynamic parameters, the adsorption process was carried out at various temperatures namely 20, 30, 50 and 80°C. A 40 mL tap water at TDS level of 1034 mg/L was allowed to equilibrate with 0.25 g sorbent. The distribution coefficient K_D, defined by equation (6), was evaluated at the specified absolute temperatures 293, 313, 333 and 353 K.

$$K_D = \frac{\text{Amount of TDS adsorbed}}{\text{Amount of TDS in solution at equilibrium}} \times \frac{V}{m} \tag{6}$$

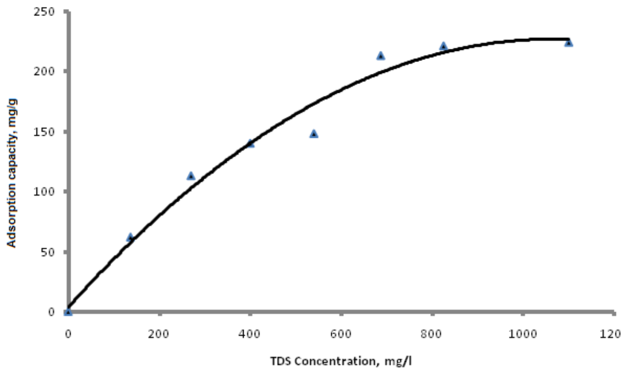

Fig. 8. Adsorption isotherm of TDS in tap water: 0.25 g sorbent, 40 mL tap water sample and 1.0 h shaking time at 25°C.

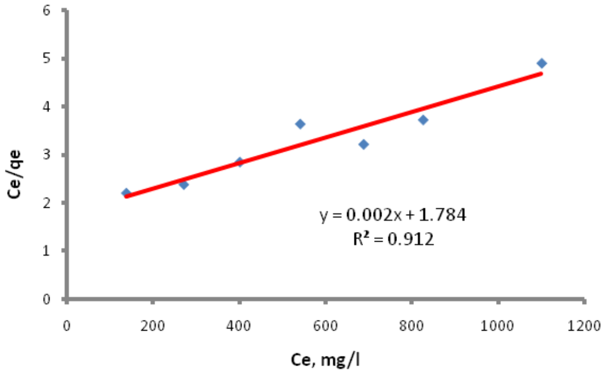

Fig. 9. Langmiur isotherm model for the removal of TDS from tap water.

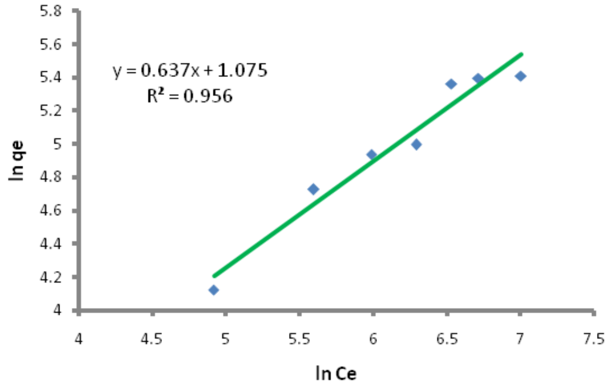

Fig. 10. Freundlich isotherm model for the adsorption of TDS onto *P. harmala* adsorbent.

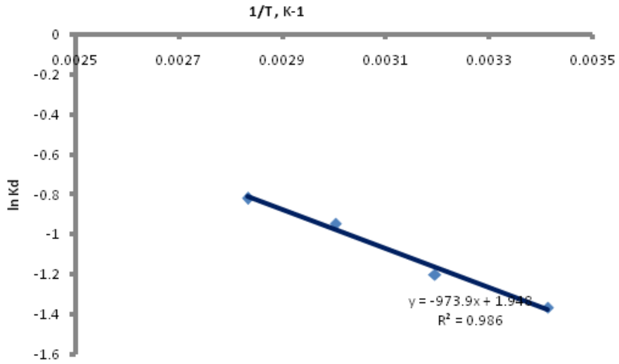

Fig. 11. Van't Hoff's plot of the effect of temperature on adsorption of TDS onto the *P. harmala* adsorbent.

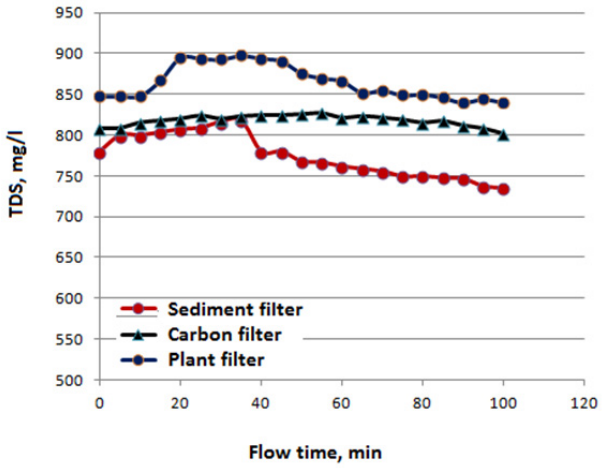

Fig. 12. Comparison of the dynamic removal of TDS using *p. harmala* packed filter versus commercial sediment and carbon filters.

where V and m are the sample volume in litre (L) and sorbent weight (g), respectively.

The Van't Hoff [36] relation correlates the equilibrium constant K_D with temperature as presented by equation (7).

$$ln\ K_D = \frac{-\Delta H^\circ}{RT} + \ Constant \tag{7}$$

where K_D and ΔH° are the chemical equilibrium constant and enthalpy of adsorption (KJ/mol), respectively. The plot of Van't Hoff equation is shown in Fig. 11. It was observed the adsorption of TDS decreases with increasing temperature. From the slope, the calculated value of ΔH° was 8.1 KJ/mol (calculated as $CaCO_3$). The positive value of enthalpy change reveals an endothermic adsorption process. Moreover, the value of ΔH° is less than 40 KJ/mol which indicates physisorption which satisfy the previous interpretation for the Freundlich multilayer adsorption. Worth mention, the attraction between the adsorbent and TDS might mainly be the electrostatic and the weak van der Waals intermolecular forces.

3.5. Dynamic removal of TDS

Under dynamic mode, tap water was allowed to pass through the fabricated *P. harmala* filter at a flow rate of 100 mL/min. The concentration of TDS in the effluent was monitored versus time. Also, the commercial sediment and carbon filters were operated under the same conditions. Comparison of the results obtained for the *P. harmala*, sediment and carbon filters are depicted in Fig. 12. Initially, very high TDS value was obtained with the three filters which might be attributed to the insufficient contact time necessary to activate the adsorption sites in the solid phase. At these early stages, typically within 20 min, the TDS increased with time and reached the maximum value. By exceeding 40 min, all filters afforded continuous and gradual decrease in TDS value. After passing tap water for one hour, the profiles remained similar while preserving the difference between them. When time reached 100 min, the TDS reached values of 840, 735 and 802 mg/L for *P. harmala*, sediment and carbon filters, respectively. Thereby, the *P. haramala* proved its worthness as an alternative filter in comparison to these commercial filters. It showed an adequate efficiency which can be considered as a valuable achievement. An inter-day investigation of TDS was carried out over 3 days. Results showed a promising enhancement of TDS since it reached a value around 690, 550 and 600 mg/L, respectively. Thus, passing tap water for a while would be expected to approach the TDS level of drinking water.

4. Conclusions

The present study investigated the quality parameters of two borehole waters and drinking bottled water. Relevant quality parameters were observed for drinking water. However, the borehole water, used as tap water, showed elevated TDS values than the recommended level. Thus, it was necessary to develop a new procedure for the removal of TDS from tapwater. The adsorption behavior of TDS in non-drinking tap water from borehole origin onto the proposed *P. harmala* sorbent was investigated. The influence of adsorption time, sorbent dose, sample concentration and temperature were undertaken. Results revealed a moderate rate of adsorption and obeyed Freundlich isotherm. The study revealed an endothermic adsorption obeyed to Freundlich multilayer adsorption better than Langmiur model. Electrostatic or coulombic forces are the main interactions between the dissolved ions and the sorbent surfaces. The outcome of adsorption and dynamic removal of TDS revealed satisfactory capability of the studied sorbent for decreasing the level of salinity which increases the benefits of *P. harmala* as an alternative and low cost sorbent for removal of TDS from water. Finally, the dynamic removal of TDS has afforded promising desalination results which should be considered for further investigations.

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