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Valorization of raw industrial waste and natural materials in the adsorption of heavy metals from wastewater of El Jadida City to be reused in irrigation

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ABSTRACT

Activated carbon as an adsorbent has been widely used to remove toxicity from wastewater, but its high cost causes restrictions in its use. For this reason, this study investigated at lab-scale the raw coal waste, namely fly ash and bottom ash, by being a potential source of pollution to be recycled and used as adsorbents. Besides coal waste, the adsorptiontes reclaim natural adsorbents materials, particularly clay soil and sea sand. The adsorbents were compound in different layers to form a filter Matrix in order to perceive the performance of those adsorbents toward heavy metals removal. The result shows that an efficient simultaneous decrease of heavy metals has taken place due to the proprieties of the filter matrix that interact with wastewater molecules and consequently enhance its quality, the highest removal efficiency of heavy metals achieved 83% for cadmium in the matrix compound of Fly ash, bottom ash and clay. The novelty of this research lies in applying raw materials with no recourse to chemical or biological treatment to the adsorbent materials. The main objective is to reclaim a low cost efficient adsorption process based on raw materials to be applied in a wastewater pretreatment plant where wastewater samples were picked out.

Keywords: Adsorption; Heavy metals; Coal waste; Clay; Sand

1. Introduction

Water is an essential source for maintaining life on our planet. Even though water is abundantly available, its quality varies systemically according to urban activity as well as industrial growth, thus, the increasing amounts of wastewater containing heavy metals, that are potentially known by being bioaccumulated, can exist long-term and are unreversed. Consequently, human health is significantly affected by most of these harmful ions even at very low concentration [1]. Recently, the Moroccan government has implemented many wastewater treatment plants to offer a significant opportunity for better discharge manage relative to stringent environmental regulations where many types of wastewater treatment plant has been explored [2]. They could be based on ion exchange, chemical settling or reverse osmosis, among them, adsorption receives considerable interest with the high efficiency in heavy metal removal [3].

Moreover, the pollution creates not only sufficient water issues, it affects also the quality of the air. Indeed, one of the most common pollution source of the air in Morocco are the thermal power plants mostly based on coal burning to produce electricity power, systemically, a new kind of pollutants residues [4], such as fly ash and bottom ash that often damages the soil and the air. In this stage, different types among coal waste have been controversial in different investigations, particularly the reuse of fly ash in cement. Likewise, previous published work had practically reclaimed fly ash and bottom bosh in wastewater purification and therefore attest their performance as adsorbents.

Indeed, it is aimed to create a sufficient environment in El Jadida city which usually faces climate and wastewater issues. However, the city relies only on a wastewater

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pre-treatment plant (WWPP) to avoid pouring wastewater into sea without prior treatment, and the plant receives daily a flow of urban and industrial liquid waste to be discharged into the sea after being submitted to several steps of pre-treatments.

In addition to the wastewater crises,the air of the city is systemically affected by industrial smoke especially those transmitted by the thermal power Plant "*Jlec*". It is located in the surroundings of ElJadida city, considered as one of the biggest thermal power plants in the country, and generates yearly 640,000 ton of waste ashes.

Accordingly, a potential solution is to use fly ash and bottom ash in the purification of the wastewater pre-treated in the plant as physical treatment based on adsorption process, then reuse it and recycle it for irrigation in the region. However, the plant is evaluated with regards to the discharge regulation but does not met with the limit concentration of heavy metals in Moroccan norm for irrigation.

Overall, the present work tested other raw materials as adsorbents in addition to coal waste to be exanimated in the adsorption process at lab-scale and then broaden the possibilities that will be adopted in the wastewater pretreatment plant. Using natural materials which have already been proven by researchers to be candidates as adsorbents [5], the raw material chosen was based on investigating locally available product materials, the use of sea sand and clay soil widespread and easily found in El Jadida city.

The present study was undertaken in order to achieve the following three objectives: 1) to evaluate the quality of the wastewater from the output of the plant through the measurement of heavy metals concentration; 2) to assess the properties and characteristics of the adsorbents materials chosen for the adsorption process namely the chemical composition, crystal structure and particle size distribution; and 3) to create at lab-scale several matrix compound of a variety of adsorbents layers. Ultimately, a performance assessment of the matrix in terms of heavy metals removal in order to achieve the irrigation regulation has been achieved [6,7].

2. Materials and methods

2.1. Sampling of wastewater

2.1.1. Description of the wastewater pretreatment plant

The region has a continental oceanic climate with a mean annual temperature of 17.4°C, and an average annual rainfall of about 372 mm. El Jadida city owns an industrial park extends on more than 117 ha on the edges of the city. The collection of the wastewater in the city is assured by many water pumping stations located in different points in the city, assessed to repress the backflow of the effluent toward the WWPP using an unitary network which is shown to be useful in decreasing the pollution generated by the industrial activities. Fig. 1 summarizes the system of the pre-treatment plant which receives a daily flow of 3,000 m^3 from wastewater.

2.1.2. Sampling of the pretreated wastewater

As it is aimed to remove heavy metals from wastewater of the WWPP, sampling points were picked out at the output of the plant,rather,after being pre-treated. Wastewater samples were previously characterized after being centrifuged in polyethylene bottles carrying out a volume of 1 L in each bottle, and stored at 4°C before analysis. The main parameters analyzed were: organic matter precisely chemical oxygen demand COD, biological oxygen demand BOD_5, dissolved oxygen, pH, ions chloride Cl^-, phosphorous ions PO_4^{3-}. The synthetic wastewater characterized was constituted of (750 mg O_2/l of COD, 875 mg O_2/lBOD_5, 213.7 mg/l of PO_4^{3-}, 1700 mg/l of Cl^-, 1500 mg/l of suspend matter, 0.94 ms/cm for conductivity and 0.1 as regards dissolved oxygen). The concentration of heavy metals contained in our samples has been evaluated. The ions that were the subject of our study are cadmium (Cd), copper (Cu), zinc (Zn), lead (Pb), arsenic (As), chromium (Cr), mercury (Hg), iron (Fe) and selenium (Se). Heavy metals analysis was carried out using inductively coupled plasma emission instrument (ICP).

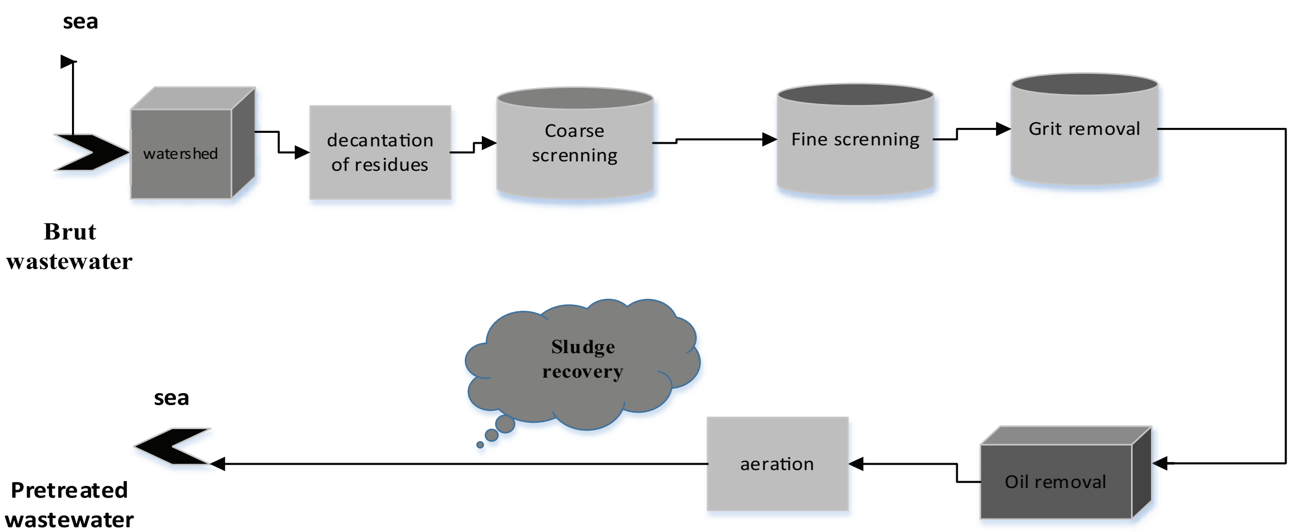

Fig. 1. The processes of the wastewater pretreatment plant.

2.2. Adsorbent materials

2.2.1. Solid industrial waste

Fly ash and bottom ash are the most common among coal wastes that are been used as adsorbents, and in this study, both ashes were extracted from the thermal power plant of Jorflasfar " JLEC", situated at a distance of 17 km from El Jadida city. Fly ash (Fig. 2a) is produced by the precipitators in the smokestacks of the coal-burning power plant. It appears in a grey color with a typical spherical particles. Fly ash is well known by its pozzolanic properties and its negatively charged surface [8,9]. Although, bottom ash (Fig. 2b) is produced by being removed from the furnace bed, sluiced to the water tank and pumped out into lagoons spread waiting to be drying up. Visibly, it appears in a dark color, their particles are quite porous and look like volcanic lava.

To improve the understanding of the filtration process, the samples of both ashes were submitted to the fluorescence X to indicate their chemical composition, then to the particle size sieving that evaluate the particle size distribution, and as regards the crystalline structure and the surface area of each type of ashes, they were involved by Taoufiq et al. [10], who has examined ashes extracted from the same power plant "Jlec" used in our experiments, thus its result would be taken into consideration during the adsorption discussion since we exhibit the same source of ashes.

2.2.2. Clay

Clay has been successfully used as an adsorbent to remove heavy metals from aqueous solutions [11,12]. The samples were collected from the surroundings of El Jadida city, where clay minerals are easily found and widespread (Fig. 3). The experimental field was established on a clay soils located in the area of *sebtouladbouaziz* far from the south of El Jadida city for 30 km.

To exhibit a particular samples in terms of texture parent material and mineralogy, samples sets used in this study was collected during the early spring 2017. Samples were divided into two parts, the first was used for the laboratory analyses, whereas the other part was used for adsorption experiments. They were stored in plastic bags at 48°C from the time of sampling until the time of analysis. For better identification of the adsorption mechanism, we have revealed information about the chemical composition of clay soil, particle size distribution and crystal structure using respectively for each analysis Fluorescence X technic, sieving and X ray diffraction technic.

2.2.3. Sand sample

Regarding sand samples, they were collected from the coastline of El Jadida city withdraw which is extended on over than 150 km, then they were carefully washed and dried at 40°C in an oven. To minutely identify the sands samples, chemical analyses were performed by atomic emission spectrometry with inductively coupled plasma (ICP-AES) and particle size distribution was investigated

Fig. 3. Visual aspect of clay soil.

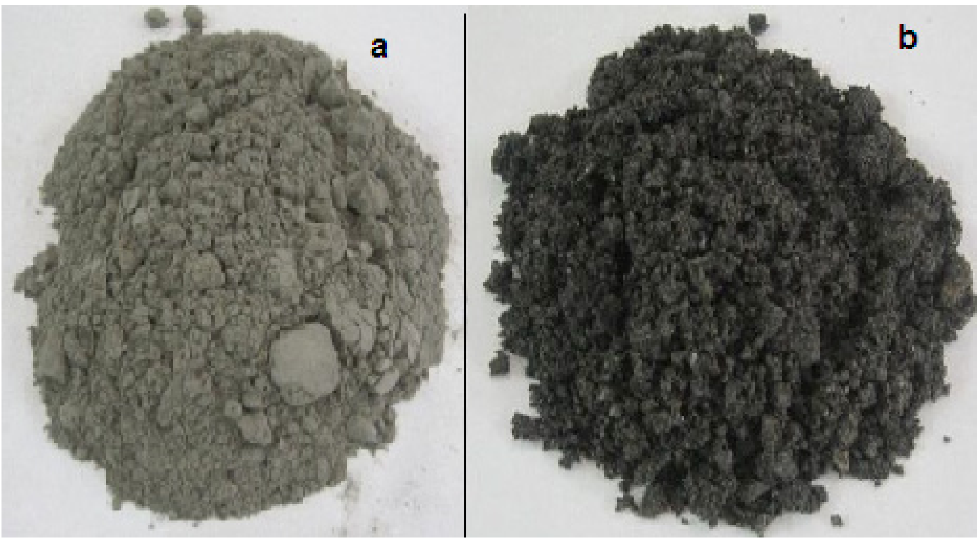

Fig. 2. Visual aspect of fly ash (a) and bottom ash (b).

by particle size sieving. Fig. 4 summarizes the localization of the sand sample.

2.2.4. Experience setup

The experiments of water purification were performed at laboratory scale, and he filtration was based on using small column as height of 120 mm and a radius of 100 mm as is shown in Fig. 5. The matrix is composed of two or three layers of adsorbents materials summarized in Table 1. This disposal has been chosen by taking into consideration the granulometry size of adsorbents materials in order to avoid clogging [13,14]. The mass of each layer of adsorbent is 50 g supporting a volume of 150 ml of wastewater to be filtered. The purification experiments were repeated five times.

3. Results and discussion

3.1. The characterization of the adsorbent materials

3.1.1. Analysis of coal ashes

3.1.1.1. Chemical composition

Chemical compositions of both ashes determined by X-ray fluorescence are presented in Table 2. It is observed that fly ash and bottom ash required approximately similar oxide content, the main components were SiO_2 and Al_2O_3 with others found at low concentrations, those components increase the capability to form to bermorite considered as an ion exchanger which has been proved its ability to adsorb heavy metals in wastewater [16]. As regards fly ash, it's been deducted that (SiO_2+Al_2O_3+Fe_2O) > 70(%), so it could be classified in the class F according to the standard ASTM C [15], it causes consequently pozzolanic activity [17].

3.1.1.2. Particle size distribution

Particle size distribution analysis is shown in Fig. 6. It can be deducted that fly ash exhibit a good correlation in

Table 1
Matrix chosen in filtration

Bww	Brute wastewater=the output of the plant
Matrix1	Sand1+fly ash
Matrix2	Sand2+bottom ash
Matrix3	Sand3+clay
Matrix4	Fly ash+clay
Matrix5	Bottom ash+clay
Matrix6	Fly ash+ bottom ash
Matrix7	Fly ash+bottomash+clay

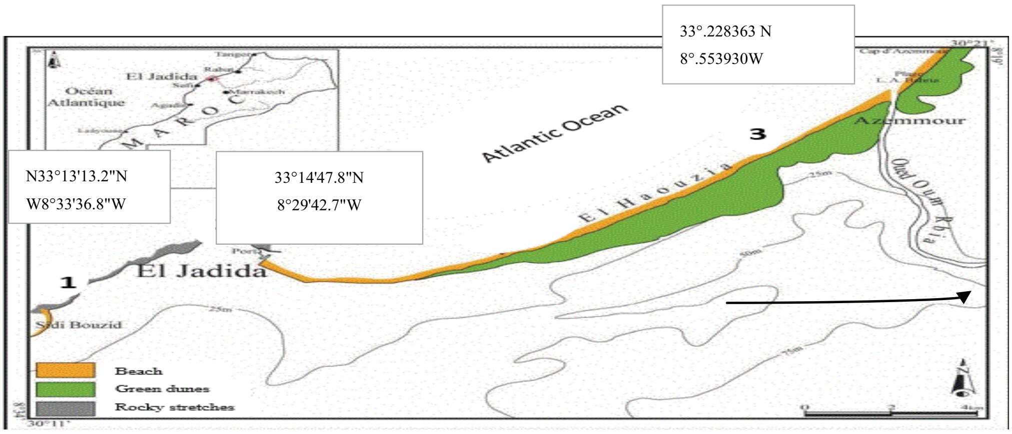

Fig. 4. Localization of the sand samples.

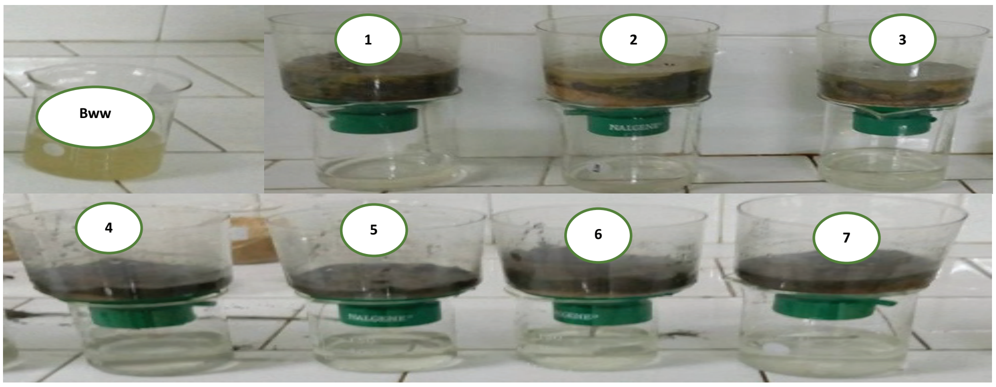

Fig. 5. The columns used in filtration.

Table 2
Chemical composition of Fly ash and bottom ash

Percent of chemical element	Fly ash	Bottom ash
SiO_2 %	57.00	52.07
Al_2O_3%	34.00	8.86
Fe_2O_3%%	3.40	8.86
CaO%	10.00	1.92
MgO%	0.02	1.09
K_2O%	1.20	1.9
Mno	0.07	0.09
TiO_2	1.24	0.98
P_2O_5	1.27	0.98

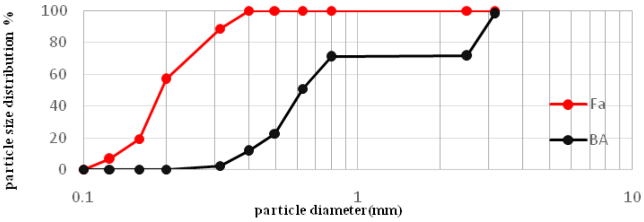

Fig. 6. Particle size distribution of fly ash and bottom as.

terms of particles falling in the range of less than 0.35 mm, thereby, it is observed a uniform distribution curve of fly ash grains, whereas bottom ash contained coarser and irregular particles due probably to the unburned coal presented in the samples and incomplete in cineration materials. The average size of bottom ash varies from fine to gravel sand (0.8 mm to 2.5 mm). The contents of CaO, P_2O_5, MgO and TiO_2 decreased as well as increasing grain size, however with increasing the contents of Fe_2O_3 and Al_2O_3 grain size decrease [18], so it is assumed that as particle size decreased, the amount of heavy metal leached from ashes increased.

3.1.2. Clay soil analyze

3.1.2.1. Chemical composition

Analysis was carried out by the fluorescence X technique to establish the chemical composition of clay soil templated in Table 3. The result shows that the soil collected to be investigated in our experiments, contains major elements of silica, quicklime alumina and iron which increase the interaction with oxides contained in coal ashes [19,20], whereas the existence of SO_3 could be explained by the industrial activity in the region.

3.1.2.2. Particle size distribution

The particle size distribution defines the texture of the soil [21], and by using a highly standard sieving, it allows a knowledge of not only the average size but also how the sizes are distributed. Indeed, the soil used in our experiments emphasize a high percentage of silt-clay sized particles passing through 2 µm, although, 50% of soil particles are ranged in 10 µm (Fig. 7).

Table 3
Chemical composition of clay soil

Oxides	Percentage %
SiO_2	15.46
CaO	9.25
Al_2O_3	5.30
Fe_2O_3	2.92
K_2O	1.26
Na_2O	0.34
SO_3	0.23

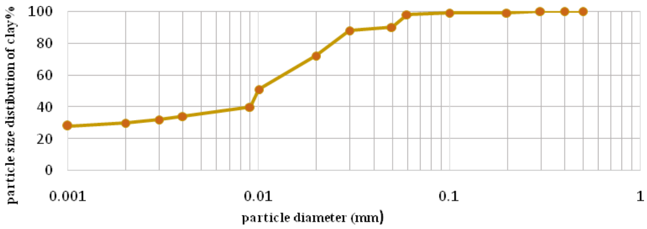

Fig. 7. particle size distribution of Clay.

3.1.2.3. Mineralogical spectrum

The mineralogical spectrum of soil epitomized (Fig. 8) was analyzed by X-ray diffractometer. The mineralogy of elements ranging in a size less than 0.2 µm fraction is dominated by illite/clay, while a major traces of quartz are excessively detected. Also some expandable minerals as dolomite, ferrouan have been revealed. In addition, noticeable reflections of Muscovite and Microcline are also present as a major accessory mineral, whereas only traces of kaoliniteare detected. The expandable minerals are characterized by the presence of a peak at 1000 coups, their presence refer to the condition to the region of sampling and the season when soils were extracted.

3.1.3. Sand analysis

3.1.3.1. Chemical composition

The chemical analysis performed by atomic emission spectrometry with inductively coupled plasma (ICP-AES) was applied to precisely indicate the chemical characteristics of the natural sand used in our experiments. The results summarized in Table 4 show that sand3 contain higher amount of alumina while sand2 is rich of quicklime compared to the other samples. The three samples are highly concentrated with silica which may allow the interaction with oxides contained in ashes to form a good layout of performant adsorbent.

3.1.3.2. Particle size distribution

The results of the sieving analysis summarized in Fig. 9 show that over 90% of the sand particles fall in the size intervals ranging between 0.5 mm and 0.7 mm. Insignificant amount of sand grains also fall in the size intervals ranging from 0.1 mm to 0.2 mm, therefore, it could be con-

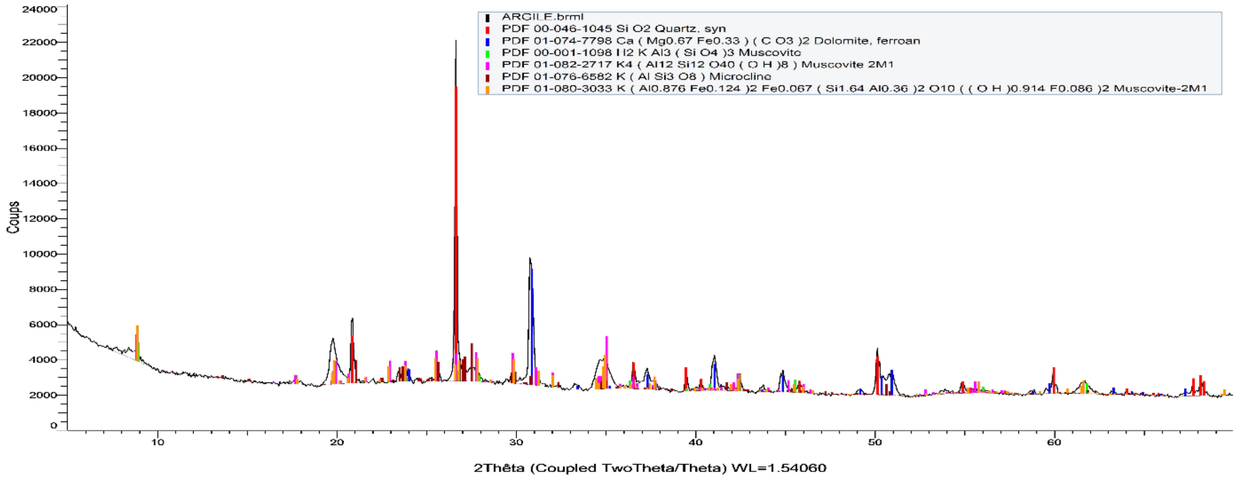

Fig. 8. X ray diffraction of clay.

Table 4
Chemical composition of sea sand (%)

	Sand1	Sand2	Sand3
SiO_2	61.14	31.69	51.54
Al_2O_3	5.16	1.73	10.77
Fe_2O_3	3.37	1.55	9.13
CaO	19.27	36.04	23.30
MgO	3.81	4.55	4.74
K_2O	1.00	0.40	0.28
MnO	0.06	0.04	0.03
TiO_2	0.45	0.28	0.03

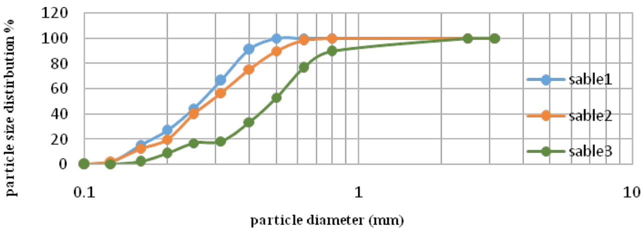

Fig. 9. Particle size distribution of sea sand.

cluded that the sand distribution in this region has significant access to fine grains [22].

3.2. *Analysis of the heavy metals concentration in the wastewater*

The wastewater collected from the output of the plant is observed to be widely accepted to be discharged into the sea, as can be deducted from Table 4. Heavy metals concentration are perfectly well below the discharge regulation except iron (Fe) and selenium (Se) whom are slightly above the norm [23]. Moreover, it is observed that the irrigation norm is not completely achieved. We found that the concentration of cadmium, mercury, iron and selenium are systemically above the irrigation regulation [24]. The

Table 5
Heavy metals concentration in brut wastewater

Heavy metals	Wastewater mg/l	Reject regulation mg/l	Reuse regulation mg/l
Cd	0.10	0.25	0.01
Cu	0.18	2.00	2.00
Zn	0.48	5.00	2.00
Pb	0.40	1.00	0.50
As	0.013	0.10	0.10
Cr	0.34	2.00	1.00
Hg	0.01	0.05	0.001
Fe	0.21	0.10	0.10
Se	0.32	0.10	0.02

presence of selenium is probably caused by the pharmaceutical industry located in the middle of ElJadida city, it could oxidize and chemically transform to water-soluble compounds and became toxic in case of high concentration [25,26].

3.3. *Analysis of the adsorption experiments*

3.3.1. *The efficiency of matrix on decreasing toxicity*

After treatment, it is deducted that all matrix chosen are candidates to achieve the irrigation regulation modeled in Table 5, due the adsorption proprieties of the materials used, namely the low electrical polarity in clay soil [27,28] which contribute to the neutralization of cations contained in the wastewater, the silica and metals oxides in ashes that gives them a high surface reactivity and the calcium silicate hydrates CSH widespread in littoral sand [29].

It is systemically observed that the adsorption of metal ions increased as particle diameter decrease, the presence of a larger number of smaller particles provides the adsorption system with a greater surface area allowing heavy metals removal and reduces also the external mass transfer resistance [30,31].

Table 6
The concentration of heavy metals after filtration (mg/l)

	Matrix1	Matrix2	Matrix3	Matrix 4	Matrix 5	Matrix 6	Matrix 7
Cd	0.01	0.019	0.009	0.006	0.008	0.005	0.005
Cu	0.008	0.0062	0.005	0.0025	0.0001	0.0005	0.0001
Zn	0.0018	0.0025	0.003	0.0087	0.006	0.002	0.001
Pb	0.0036	0.002	0.00125	0.0013	0.0010	0.0012	0.003
As	0.00812	0.009	0.00633	0.0049	0.0055	0.003	0.005
Cr	0.02	0.021	0.03	0.012	0.0078	0.006	0.0098
Hg	0.0025	0.008	0.0069	0.0025	0.008	0.0001	0.0001
Fe	0.06	0.06	0.065	0.0089	0.01	0.008	0.009
Se	0.0078	0.0087	0.0068	0.0056	0.0062	0.007	0.0052

Another interpretation for our results was confirmed by Erdem et al. [32], who proofed that heavy metals adsorption is attributed to different mechanisms of ion-exchange processes, and during this process, metal ions had to move through the adsorbent matters, and they had to replace exchangeable cations (mainly sodium and calcium), therefore, he indicates that energetically less favorable sites become involved with increasing metal concentration in the aqueous solution. Xiaofuet al. [33] have also reported in the same concept that adsorption efficiency of metals increase with decreasing metal initial concentration, which is the case in the present work, as can be seen, all initial concentrations of metals are less than 0.5 mg/l. Furthermore, Ashtoukhy et al. [34] explained that at low concentrations, metals are adsorbed by specific sites, while with increasing metal concentrations the specific sites are saturated and the exchange sites are filled leading to less adsorption efficiency.

It is observed, that there is a significant reduction in the adsorption of cadmium (Cd), copper (Cu) and lead (Pb) in the matrix compound of clay, this is explained by the interaction between the low electrolyte concentration in clay and the high charge density contained in Cd, Cu and Pb ions [35], also it has been proven that increasing ionic strength made the potential of the adsorbent surface less negative and thus would decrease metal ion adsorption.

In regards to the reduction of iron (Fe), it is due to the interaction between CaO present in fly ash and SiO_2 present in the sands to form calcium silicate hydrates C-S-H. This latter can incorporate with Fe ions by substitution.

The matrix 7 contributes to the highest Zinc (Zn) reduction which can be explained by the adsorption capacity of the bottom ash and fly ash. So it can be incorporated in the interlayer spaces of the calcium silicate hydrates C-S-H.

For chromium (Cr), the highest abatement is achieved in the matrix 6, which is due to the fact that fly ash and bottom ash have a high surface area and great amount of titratable functional groups, thus, the creation of unclenching force between the accessible sites of both ashes and Cr ions [36,37].

Mercury (Hg) is also an important parameter to test the quality of water, as it is shown, mercury (Hg) exhibits a greater affinity towards fly ash in matrix 4,matrix6 and matrix7. This is elucidated through the fact that the sites of Fly ash are accessible to the Hg^0 molecules [38].

Concerning Selenium (Se), the highest efficiency is recorded in the matrix compound of ashes, this is due to the fact the major species of Se interact with iron in Fly ash and bottom ash [39].

4. Conclusions

The present study investigated the characteristics of several adsorbent materials potentially known and proven by other researchers to be candidates in wastewater purification, particularly, sea sand, clay soil and coal waste, with the aim to involve a simultaneous adsorption process. This work explored the result of several proprieties of adsorbents to minutely perceive the mechanism of the adsorption setted up in our experiments. Some characterization was deducted from other studies that has previously investigated fly ash and bottom ash picked out from the same source, namely the surface area of ashes [10].

The global objective is to recycle the wastewater recovered from the wastewater plant and then reuse it in irrigation. The experience was set up at lab scale to evaluate the matrix compound of different layers of sand, soil, fly ash and bottom ash. Consequently, it is shown that all matrix was shown to be candidates regarding the respect of the Moroccan irrigation for heavy metals removal. The result of this experience was extensively satisfactory so the idea to carry out the filtration in larger scale and adopt it in the wastewater pretreatment plant such as the plant M'zar in Agadir city in Morocco [40].

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