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Characterization of urban wastewater and elimination of their pollution using clay – materials in the M'zab Region, Algeria

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ABSTRACT

The wastewater discharges are increasing due to industrialization and rising in the standard of living of the population in the M'zab region. This study is interested in the treatment of urban wastewater with raw clay. Wastewater treatment requires a series of steps involving physical, physico-chemical and biological treatments. Among the largest wastes present in wastewater, the treatment must at least eliminate the major part of the carbon pollution. Clays are characterized by a high capacity for adsorption, ion exchange, and swelling as well as by a particular rheological property. According to the results obtained, the treatment of urban wastewater in the M'zab valley with clay is effective in the adsorption of mineral salts of calcium, sulfates, chlorides, etc... is due to the fixing of the particles on the clay. This treatment shows an efficiency of elimination of organic matter, obtained for BOD_5 with a yield of 90% after 9 d of contact for iron reaches 100% and one notices a decrease in the COD as well 60% as of other parameters of pollution. Indeed, we notice that the kinetics of adsorption of the purified clay presents the best capacity than the raw clay arrives up to 82% for nitrates and 95% for orthophosphates. Wastewater treatment will not only reduce the cost of treatment but also contribute to the preservation of the environment.

Keywords: Adsorption; Clay; Kinetics; Treatment; Wastewater

1. Introduction

Global water availability is unchanging [1,2]. Since his existence on earth, man has always used the same water; this is why water, for a long time freely available in many parts of the earth, is today seriously threatened by what is called pollution [1,2]. The hydrosphere degradation is the result of discharges of wastewater without treatment or at an insufficient level of treatment [3]. The problem is even more serious in the case of industrial effluents which are toxic in nature. In general, the effluents require a treatment, more or less important according to the degree of deterioration of water, before their rejection in the natural environment. The existing processes are numerous: physical, chemical or biological [4]. The choice of a process for the treatment of discharges depends on a number of factors, the most significant of which are: the composition of the effluent and the type of reuse [3]. Industrial and domestic wastewater contains many organic and inorganic pollutants that are hard to remove using other common techniques (precipitation, chemical precipitation, adsorption and membrane separation) [4]. These pollutants are found in very small concentrations that which makes their depollution difficult [5]. They essentially require advanced

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treatment technologies to have a final effluent that complies with the national and international standards. In addition, strict regulations have been proposed to handle, treat, and reuse this heavily polluted wastewater [6]. But to solve the problem of micropollution, several researches are oriented towards the adsorption process which is based on the use of adsorbents such as the best known, activated carbon. Further, research interests are given in recent years to the study of clays [5,6] as adsorbent. This interest is justified by their abundance in nature, the size of the surfaces they develop, the presence of loads. Many recent scientific works [7–10] relating to the purification of wastewater by adsorption on these new solid supports (prepared from the mixed intercalation of clays by pillars generally hydroxymetallic and/or surfactants) report information on the different methods synthesis, characterization and application [11].

Adsorption on raw and modified clay constitutes one of the most important physicochemical processes for the removal of most organic and inorganic micropollutants. A large number of studies [12] have highlighted the interest of using clays for water treatment and however underline the influence of various parameters. Clays are characterized by a high capacity for adsorption [11] ion exchange and swelling as well as specific rheological properties. Clay modification/activation is a process that consists of improving the adsorption properties by subjecting it to a thermal or chemical treatment [11,13].

The aim of this study is to prepare a material based on natural clay to apply it as an adsorbent in the treatment of urban wastewater. The natural adsorbent material is modified by chemical treatment with sulfuric acid.

2. Materials and methods

2.1. Experimental protocol and sample preparation

The assessment of the physicochemical quality of groundwater in the hills was possible using water sampling taken during a campaign in May 2021. Thus, the physicochemical parameters measured in situ and at the laboratory.

The study was carried out primarily to characterize the urban wastewater from the M’zab valley treated with clay from Bouhraoua ward located 600 km southwest of the capital of Algeria for periods at 3 d intervals. Three samples A, B, and C were taken on the 3rd, 6th, and 9th day, respectively, during the flow of the Wadi M’zab before entering the station wastewater treatment plant (WWTP) Fig. 1 shows the spatial distribution of the sampling points.

The clay which raw and purified were the materials utilized in this treatment, 21 samples were prepared, 9 of which were treated with raw clay, 9 were treated with purifying clay, and 3 samples of raw wastewater were used as a negative control. The raw clay used in this work is an illite-family green clay with a mass of 50 g that was crushed and sieved to remove impurities.

The dosage of quality physicochemical parameters was carried out according to standard analytical methods [14,15]. The parameters studied, the methods, and the analytical material used for clay analysis are presented in Table 1.

2.2. Activated materials

2.2.1. Purification of raw materials

Purification is a process that consists of improving the adsorption properties of clay by subjecting it to thermal and chemical treatment.

The purification of the clay by different chemical processes (sodium citrate, hydrogen peroxide, sodium chloride, etc.) and a physicochemical characterization allowing to have an active sample that is easy to adsorb organic micropollutants.

2.2.2. Operating protocol for obtaining purified clay

To eliminate pebbles or other impurities, the clay is crushed and then sieved through a mesh sieve with a 2 mm diameter [16]. Our clay-based materials have been tested in the treatment of urban wastewater discharge and have proven to be effective [17].

2.2.3. Destruction of unwanted material

The carbonates are eliminated by relatively dilute (HC1N/10)

Fig. 1. Presents the sampling points A, B, C.

Table 1
Parameters studied and the methods of analysis

Clay analysis	Methods
Granulometric analysis by dry-drying after washing	NFP 94-056 (March 96)
Granulometric analysis by sedimentation	NFP 94-057 (May 92)
Atterberg limits	NFP 94-051
$CaCO_3$	NFP 15-461
Insoluble, %	NFP 15-461
SO_3^{-2}	BS1377
Water content and density	NFP 94-050 and NFP 064
Water analysis	Methods
BOD_5	Méthode instrumentale OxiTop, WTW AFNOR T 90 105
COD	Titrimetric method ISO 6060
Orthophosphates	Orthophosphates molecular absorption spectrophotometric ISO 15681
Nitrite (NO_3^-)	Molecular absorption spectrophotometric ISO 7890
Nitrogen ammoniacal (NH_4^+)	Spectrophotometric method ISO 7150/1
X-ray diffraction	PANalytical X'Pert PRO with ultra-fast detector X-ray tube anode material: Cu

The carbonate constituents are generally calcite and dolomite. Acidify 200 g of raw clay in 1 L of distilled water for about 12 h until the pH is between 3 and 3.5, allow it to settle then bring the pH back to around 7 with distilled water.

2.2.4. Sedimentation and recovery of clay

After the operation to destroy the undesirable material, the clay is dispersed in a molar solution of NaCl 40 g/L for 24 h, the clay is washed by centrifugation between 4,000 and 5,000 Tr.

The last washing operations (until negative silver nitrate test). The clay was freed from chlorides, dried at 60°C then ground. It is put back to dry this time between 80°C and 100°C. To get ready for the test, a second fine and desirable grinding is homogenized the clay powder.

Take a quantity of 10 g of raw clay in 200 mL of wastewater and the same for the purified clay and put them in a Jar Test shaker at 150 tr for 2 h then put them in the centrifuge at 4,500 tr/min for 15 min.

The purification performances were evaluated according to the following formula [18].

$$\text{Abatement}(\%) = \frac{(C_E - C_S)}{C_E} \times 100$$

where C_E — concentration of raw water in mg/L and C_S — concentration of treated water in mg/L.

3. Results and discussion

3.1. Characteristics of the clay used

According to the granulometric study, clay is characterized by a great adsorbing power due mainly to its large specific surface area which makes. The binding of heavy metals is becoming more and more important.

Table 2
Physical characteristics of sampling of clay

	Sample 1	Sample 2
Depth, m	2.3–2.7	2–4.6
Gravel, %	2	6
Coarse sand, %	6	2
Fine sand, %	18	16
Silt, %	64	46
Clary, %	10	30
Dry density, T/M^3	1.32	1.44
Wet density	1.8	1.9
Water content, %	36	32
Degree of saturation, %	94	99

Table 3
Chemical analysis

Insoluble (%)	Carbonates (%)	Sulfates (%)	Observation
71.5	12	4.66	Gypsum clay
76.5	06	4.97	Gypsum clay

Chemical analysis of some types of clay, is as follows (Tables 2 and 3).

Based on the particle size analyses performed by dry sieving after washing and by sedimentation, we produced the graph presented in Table 2 which demonstrates that our clay was composed of 10%–18% fine sand, 46%–64% silt, and 10%–30% clay.

Moreover, the following tests determining the water content and the Atterberg limits resulted in liquid limits of 47% and 79%, plastic limits of 22% and 44%, and plasticity indices of 23% and 35%. These results allowed us to establish the Casagrande abacus, which classified our clay as plastic and gypsum clay.

NB: It should be noted that the particle size analysis was only carried out on a fraction less than 10 mm.

3.2. Characterization of clay by X-ray diffraction

The spectra obtained indicate that this clay is well crystallized and the peaks are narrow and rectilinear. The X-ray diffraction patterns of the sample are dominated by quartz [SiO_2] and halloysite [$Al_2Si_2O_5(OH)_4$] [17].

The XRD powder diffraction pattern of clay and solid samples are shown in Fig. 2. The recorded XRD pattern of clay is typical of clay varieties, where all peak positions and relative intensities match perfectly the reported data [19–21], indicating that the product is highly crystalline and free from impurities. The basal spacing d_{001} of clay is 0.715 nm.

3.3. Physico-chemical characteristics of water

The results of the analysis of urban wastewater show the existence of pollution characterized by a high organic load [22,23]. The interpretation of the results is based on the results of 9 d of treatment with raw clay and purified.

3.3.1. pH

Measuring the pH of wastewater indicates the alkalinity or acidity of the wastewater pH 7.40–7.70.

For water treated with clay, the pH values were narrowly between 7.9–8.20 (Fig. 3).

We also observe a disturbance of the pH at some points with the contact time. The adsorption of organic matter and certain minerals on the surface of the clay leads to the basicity of the medium, that is, to an increase in pH, for example, point A pH 7.9, and an increase in the pH, especially at point C pH 8.11 and B pH 8.20, the medium becomes basic with clay purify.

3.3.2. Electrical conductivity

With the clay treatment, the EC value remains almost stable during the three and 6 d 6,040 µS/cm. A decrease in the value of the conductivity is observed with the contact time of 9 d (6,000–6,013 µS/cm) for clay raw and (5,900–5,831 µS/cm) for clay purify in point A (Fig. 4).

The adsorption of cations and anions on the surface of the clay is probably the cause of this decrease.

We find almost the same results we can conclude that the contact time is insufficient to have good results, which explains the effect of time on the phenomenon of adsorption [24,25] at the surface of the clay is important. Raw and purified clay treatment does not show a large variation in conductivity.

3.3.3. Turbidity

Among other things, it was found that the turbidity was greatly reduced by the use of clay. The turbidity values of the wastewater decreased to a value of 0.1 NTU with very high yields which are of the order of 100% (photos 1), respectively given by raw and purified clay. This explains the importance of the effect of temples on the phenomenon of adsorbents at the surface of the clay. The decrease in turbidity is therefore due to the interception and fixation of the particles on the clay.

3.3.4. Suspended maters

The amount of suspended matter in raw wastewater was around 6,460–8,060 mg/L. The concentration of dry residue varied between 5,310 and 5,512 mg/L for the treatment with raw clay abatement of 17% and between

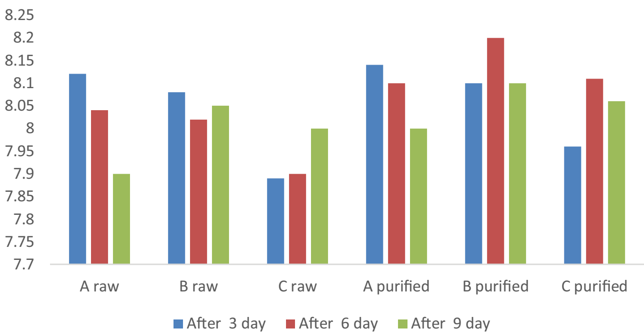


Fig. 3. Variation of pH after treating by clay (raw/purified).

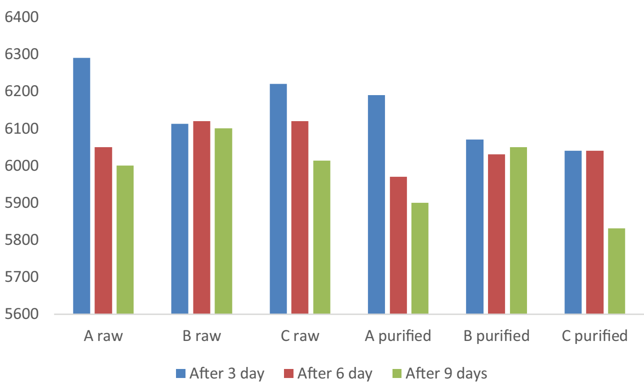


Fig. 4. Variation of conductivity after treatment by clay (raw/purified).

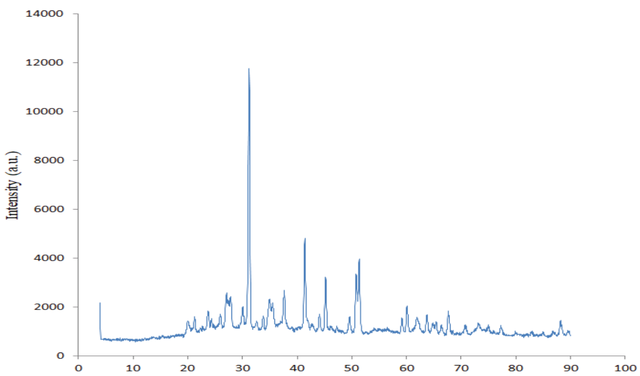


Fig. 2. Characterization of clay by X-ray diffraction.

5,200–5,400 mg/L with purified clay with abatement of 19%. Removal efficiency increases. So it is higher after the third day (Fig. 5).

3.3.5. Mineral element

3.3.5.1. Calcium (Ca^{2+}) magnesium (Mg^{2+}) chlorides (Cl^{-}) and sulfates (SO_4^{2-})

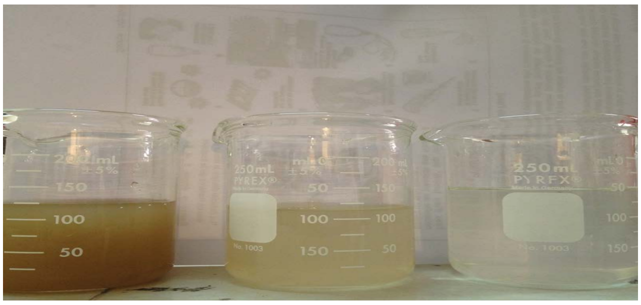
The presence of Ca^{2+} ions in the water indicates either a dissolution of the carbonate formations, calcite ($CaCO_3$), dolomite [(Ca, Mg)CO_3] or the dissolution of evaporitic formations, gypsum [$CaSO_4$, 2(H_2O)] and anhydrite ($CaSO_4$).

Magnesium is one of the most widespread elements in nature, it constitutes about 2.1% of the earth's crust, and Ca^{2+} and Mg^{2+} ions form the hardness of the water.

A decrease in the Ca^{2+} values is observed with time for raw and purified clay.

With the abatement of 43% and 60%, respectively, the same results as Mg with a reduction of 13.18% and 25.8% for the two raw and purified materials for 9 d, remember that these cations follow the same directions. The concentration of chlorides and sulfates varies between (551.61–725.11 mg/L) and (1,970–2,216 mg/L), respectively before clay treatment, removal efficiency increases. This makes it higher after 9 d, with an abatement of 13% and 26%, the same results as sulfates with a reduction of 21% and 50% for the two raw and purified samples the reduction of chlorides and sulfates are due to the fixing of the particles on the clay.

We also find that the treatment with raw clay and treated clay shows a large significant variation with the minerals showing the efficiency of the clay purifying with a yield reaching 50%.



Photos 1: Variation of Turbidity after treating By clay (raw/purified) (photo .Z. BABAAMER).

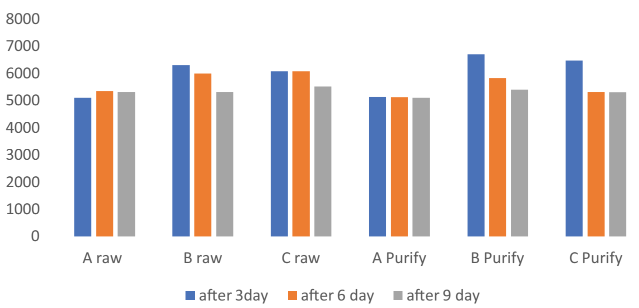


Fig. 5. Variation of suspend maters after treating by clay (raw/purified).

3.3.6. Elements of pollution

The elements of pollution are negligible in wastewater due to the high mineralization.

3.3.6.1. Nitrates NO_3^-

The concentration of nitrates varies between (18–36 mg/L), respectively after clay treatment with abatement of 55% and 82% for purified clay after 9 d, this value does not exceed the WHO standards.

Indeed, we notice that the adsorption kinetics of the purified clay has the best capacity than the raw clay.

3.3.6.2. Ammoniacal nitrogen (N–NH_4^+)

The concentration of ammoniacal nitrogen given by wastewater was between 1.5–2.9 mg/L and ranged between 0.28 and 0.72 mg/L for the raw clay and 016 and 0.64 mg/L for clay purified. The values obtained meet the standard for water intended for irrigation according to FAO (2003) ($NH_4^+ < 0.2$ mg/L) [26] (Fig. 6).

3.3.6.3. Orthophosphates (P–PO_4^-)

The average results of treatment by the two types of clays are (0.7–0.84 mg/L). These levels meet the thresholds set by the WHO (0.94 mg/L) for irrigation water [27,28].

According to the raw clay treatment results, the orthophosphates reduction is 90% and 95% for clary purify and we got good results after the first day of treatment, so the decrease in ammonium ion content could be explained by the effectiveness of this material or the good oxygenation of the water at the time of agitation leading to the oxidation of ammonium, but some disturbance is observed due to the presence of organic matter.

3.3.6.4. Biochemical oxygen demand and chemical oxygen demand

The BOD_5 value for wastewater is between 165–180 mg·O_2/L, this value remains higher than the specific discharge limit value (120 mg/L·O_2). The decrease in the biochemical oxygen demand is remarkable after the second and third day of treatment with yields varying

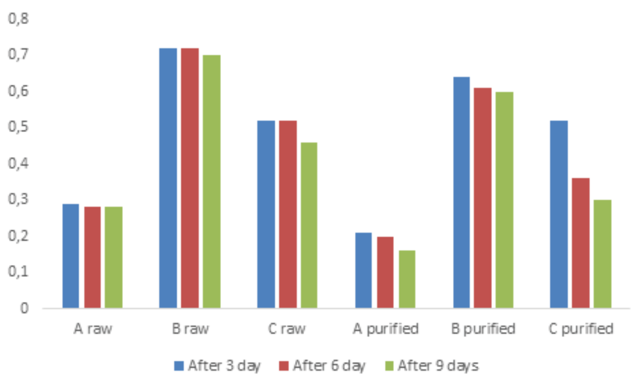


Fig. 6. Variation of ammonium (NH_4^+) after treatment by clay (raw/purified).

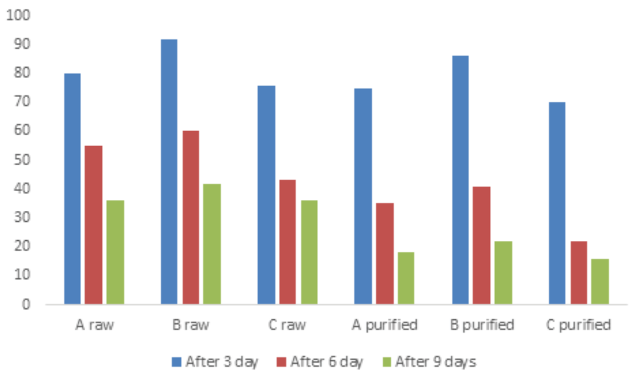


Fig. 7. Variation of BOD_5 after treating by clay (raw/purified).

between 78% raw clay and 90% for purified clay after 9 d, the decrease in BOD_5 due to the adsorption of organic matter [29–31] (Fig. 7).

The amount of COD in wastewater is between 486–546 mg/L·O_2, this value remains higher than the specific discharge limit value (300–1,000 mg/L·O_2) The COD values show a non-negligible variation during the days of clay treatment, and the COD value increases after the first day of clay treatment the increase in this is due to the presence of the organic matter, the origin of the latter is the clay itself, in addition, it decreases in the second and third day and gives a yield of 60%, we can conclude that the contact time is important to have good results, which explains the effect of time on the phenomenon of adsorbents.

The kinetics of adsorption during the first days of reaction can be interpreted by the fact that at the start of adsorption, the number of active sites available at the surface of the adsorbent material is much greater than that of the sites remaining after a certain time.

The results of the analyses indicate that the coefficient of biodegradability is in the range of $2.15 < COD/BOD_5 < 2.3$ in all samples, so the natural purification process is not effective.

If the mineralization indices show the above-mentioned influences, those of organic pollution (BOD_5, COD, and O_2) give an idea of the organic load released. The spatial profiles of BOD and COD are inversely proportional to that of dissolved oxygen.

3.3.6.5. Iron (Fe^{2+})

Low concentrations of iron (0.83–1.08 mg/L) obtained by wastewater analyses are noted. According to the raw clay treatment results, the iron reduction is 100% and we got good results after the first day of treatment.

The treatment by raw clay and treated clay shows a large significant variation shows the effectiveness of purifying clay with a yield reaching 100%, especially with the elements of pollution exp iron.

By comparing our results, we find that the majority of the values are lower than that of the initial values before treatment, and we deduce that our method of treatment with clay and specially purified clay is effective.

Based on the analysis of the results obtained in this study, the following assessments can be made:

This order of distribution of elements is practically.

The order of abundance of cations (mg/L) obtained in these waters is as follows:

$Na^+ > Ca^{2+} > Mg^{2+} > K^+ > NH_4^+$

And of the anions:

$SO_4^{-2} > Cl^- > HCO_3^- > NO_3^- > PO_4^{3-}$

retains within all sampled points.

This method of treatment is simple and effective, inexpensive, does not require sophisticated installations, and without adding chemicals compared to the technical method which uses at the level of the WWTP and has no risks to the environment.

4. Conclusion

The study was carried out primarily to characterize the raw effluents and the water treated with raw clay. The monitoring concerned the physico-chemical parameters to authorize or discourage the reuse of treated water for irrigation. Clay in the southern region is a very abundant raw material, its use in the purification of wastewater will allow us not only to reduce the cost of purification but also to contribute to the preservation of the environment.

The particle size analyses carried out by dry sieving after washing and sedimentation, show that our clay is composed of 10%–18% fine sand, 46%–64% silt, and 10%–30% clay. These results allowed us to establish the. Casagrande abacus, which classified our clay as plastic and gypsum clay.

The results obtained indicated that the treatment of urban wastewater from the M'zab valley by clay is given good results concerning the adsorption of mineral salts of calcium, sulfates, chlorides, etc.

The reductions in the mineral salts are observed and explained by the attachment of the looms suspended on the surface of the clay. As well as the best capacity the raw clay arrives up to 60% for calcium and 50% for sulfates.

The adsorption of nitrites and ammoniums is maximum, which represents an elimination rate of 100% so the adsorption of unwanted elements is greater than the salts. This treatment showed an efficiency of elimination of organic matter, obtained for BOD_5 with a yield of 90% after 9 d of contact. Indeed, we notice that the kinetics of adsorption of the purified clay presents the best capacity than the raw clay arrives up to 82% for nitrates and 95% for orthophosphates. The best elimination of COD we have obtained by the treatment with clay during the 9 d with a yield of 60%, this estimate of the yields we concluded the effect of time on the phenomenon of adsorbents at the surface of the clay is important.

It is also possible to increase, in practice, the volume of water to be treated by adding quantities of suitable active clay, in a static or dynamic process of adsorption to replace treatment plants.

The result of the physicochemical analysis shows that the treatment of wastewater by natural processes is more effective, as a result, gives a quality of treated water used for irrigation.

We are also supporting the study of reinforcement treatment facilities by an adsorption step to reduce pollution rates in wastewater.

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