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Research Article

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Removal of bacteria by adsorbents prepared from moroccan oil shale

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ABSTRACT

In this communication, we present the results of a study on the process optimization for the development of adsorbents from oil shale Moroccan of Timahdit layer (Y) and their application to the elimination of certain types of bacteria contained in the domestic liquid wastes. The adsorbents prepared thermally are characterized by different physico-chemical techniques and their performance adsorption is evaluated by determination of the maximum adsorption capacity of methylene blue. Bacteriological tests were performed on bacteria, Escherichia coli type, contained in domestic liquid wastes. The characterization results showed that the adsorbent obtained by thermal activation of the decarbonated raw rock temperature equal to 300 °C for 2.5 hours in air has good textural and structural properties. In addition, the tests of elimination of bacteria (Escherichia-coli) are conclusive, and allow providing a broad use of these adsorbent in this type of application.

Keywords: *Oil shale; Activation; Adsorbents; Adsorption; Bacteria.*

INTRODUCTION

Environment preservation, in particular, the problem of water pollution is a major preoccupation portaged by all public, industrial, scientists, researchers and politics deciders at national and international levels. For many years, researchers have interested to develop methods of treatment of waste water containing organic or inorganic substances stable or radioactive. The techniques commonly used in the case of liquid effluent treatment are: ion exchange, coagulation [1-2], the biological treatment [3-4], or the adsorption of various organic, minerals, natural or synthetic materials [5-6]. Many research works were then directed to methods of treatment using natural materials for their abundance and their low cost, among them we can include clay minerals [7-8], zeolites [9-10] and various carbonaceous adsorbents [11] prepared from abundant natural materials and minor, products of some industries or agricultural waste [10-13].

In this context, we set a goal for this work enhancement a local material which is very abundant in several national sites, through its transformation into adsorbents; it is Timahdit's Moroccan oil shale. Two precursors were subject of this study, a powder obtained directly from the raw rock layer (Y), the second precursor (YH) resulting from the decarbonation of the raw rock.

In this paper, we present results on the preparation and characterization of adsorbents and their use in testing the elimination of bacteria species Escherichia coli.

EXPERIMENTAL SECTION

Preparation of adsorbents

The adsorbents used in this study were prepared from Moroccan oil shale of Timahdit, this deposit is located in the Middle Atlas region 35 km south of the town of Azrou spread over an area of 100 km^2. It consists of a vein of shale from 100 to 150 m thick [14-16]. In this rock, the organic matter is embedded in a mineral matrix consisting mainly of carbonates which are associated with other species, such as silicates, free silica and pyrite. Table 1 summarizes the mineralogical composition of this layer Y.

Table 1 Mineralogical composition of the Timahdit oil shale [15]

Constituent	Calcite	Dolomite	Clays	Silicate	Organic matter
Wt. (%)	15.16	12.33	26.87	21.75	23.89

Preparation of precursor

Samples of the raw rock (Y) were crushed and ground to a fine powder with a particle size <0.08 mm. A given amount of this powder was added, with stirring, a solution of 6N HCl until complete release of carbon dioxide. Reactions that result from this process are:

$$CaCO_3 + 2HCl \longrightarrow CO_2 + CaCl_2 + H_2O$$

$$CaMg(CO_3)_2 + 4HCl \longrightarrow 2CO_2 + CaCl_2 + MgCl_2 + 2H_2O$$

After filtration, the solid residue was thoroughly washed in a Soxhlet apparatus, using water as a solvent to remove traces of acids and stabilize the product. The resulting product, referenced by YH, is dried in an oven for 12 h at 110 °C and stored for a possible future use.

Thermal Activation-Y and YH

For thermal activation of Y and YH, samples of each precursor is treated in a furnace under air at different temperatures ranging from 200 to 450 °C, the treatment time is between 0.5 h and 3 h. The choice of these temperatures is dictated by the large changes known structure of the organic matter contained in the precursor in this temperature range [14-16]. Before being used in adsorption tests, the prepared adsorbents, named by Yax YHAx where x is the activation temperature, are characterized by different physicochemical techniques to reveal the effect of activation at their textural and structural properties. The techniques used were the surface area (by BET method), the surface functions (by the method of Boehm) and the maximum adsorption capacity of methylene blue.

Antibacterial tests adsorbents prepared

Escherichia-coli are a germ of normal intestinal flora of all animals, including humans. The presence of this microorganism in water consumption is a significant risk to the health of those who consume it [17].

To test the ability of our adsorbents to eliminate the Escherichia coli, we emerged a mass of 1g of each adsorbent in two syringe of capacity equal to 10 mL, equipped with a filter paper disc in their lower end to avoid the eventual leaks from the adsorbent during elution. The syringes are then wrapped in aluminum foil and sterilized in an autoclave at 120 °C for 20 min.

1 mL of a 24 h culture Escherichia-coli is eluted through the different adsorbents. Elution is performed with moderate pressure on the plunger of the syringe with your thumb. In order to obtain a satisfactory elution, washing with 4 mL of physiological water freshly prepared and sterile is made immediately after elution.

RESULTS AND DISCUSSION

Properties of adsorbents used in the tests

Testing bacteria removal is performed on the two adsorbents YA and YHA representing better adsorbents obtained by heat-activation of precursors Y and YH at a temperature equal to 300 °C for 2 hours for the first and for the second 2.5 h. Table 2 summarizes the results of the characterization of the two adsorbents by three different analytical techniques.

Table 2 Characterization of two prepared adsorbents YA and YHA

	groups surface fonctions (méq/g)			Total (méq/g)	Q_{max} en BM (mgg^{-1})	S_{BET} (m^2g^{-1})
	carboxylic	lactone	hydroxyl			
YA	0.28	0.24	0.18	0.72	145	32
YHA	0.40	0.36	0.26	1.02	175	85

The values in Table 2 show the importance of the maximum adsorption capacity of adsorbents YA and YHA despite the low values of their specific areas. This finding can be justified by the development of surface features that play a very important role in the adsorption process. However, it should be noted that this value differs from one sample to another 147 mg / g for YA and 175 mg / g for YHA. This difference can be explained by the rate of organic matter present in each precursor and the beneficial effect of thermal activation in an oxidizing atmosphere (air). This type of activation on the one hand favors the formation of a carbon skeleton rich with high organic carbon, and on the other hand the development of its porosity, for the release of light molecules (CO and CO_2) formed by controlled oxidation of the organic matter existing in the precursor. These changes can be identified by examining the morphology of these products. Indeed, the morphology, taken by scanning electron microscope (SEM), of the precursors (Y and YH) and the adsorbents (YA and YHA)

(Figure 1) indicate that these structures are more developed porosity and well distributed over all samples.

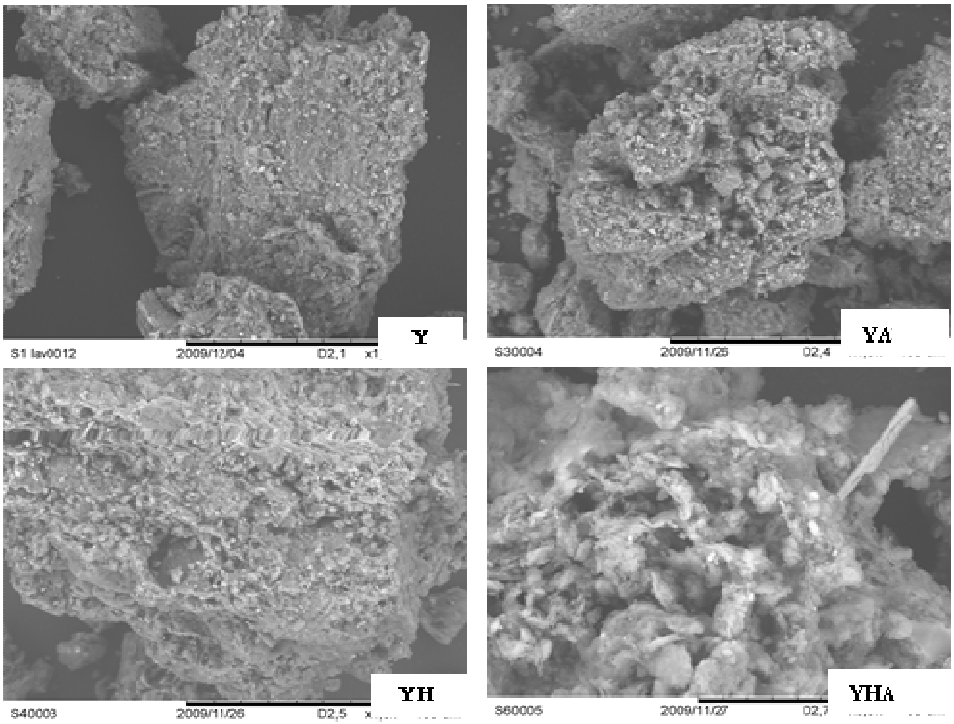

Figure 1. SEM of Y, YA, YH and YHP.

Test elimination of germs bacteria (Escherichia-coli)

Generally carbonaceous adsorbents are used for industrial purposes, in particular the treatment of sewage by various pollutants. For this purpose we conducted tests to eliminate germs bacteria very answered in domestic wastewater (Escherchia-coli) and compared the results with those obtained by the use of activated carbon sold for this purpose.
The results of these tests are summarized in Table 3, the examination of values shows the efficacy of adsorbents prepared vis-à-vis the bacterium Escherichia coli. Indeed, from a solution containing 74,107 CFU/mL, we did not detect any bacteria in the petrie box at the end of tests. It appears through these results that these adsorbents are better than some commercial adsorbents used in water treatment.

Fixing bacterial germs is surely the result of complex interactions between the microorganism, in particular, the cell wall and the adsorbent [17-19]. It appears that the surface functions, developed on the surface of the adsorbents, were participated to the removal of virtually total microorganisms either by the heterogeneity of the surface, or by the presence of divalent cations or by groups of acid C-OH, C=O, C-OOH in the vicinity [19].

Table 3 Results bacteria counts wastewater CFU/mL

adsorbent	Stock solution **UFC /mL**	Number of bacteria **UFC /mL**	references
YA	74.10^7	0	this work
YHA	74.10^7	0	this work
CAC(1)	54.10^7	75.10^4	[15]
CAC(2)	28.10^9	82.10^6	[16]

CONCLUSION

Tests to eliminate bacteria species Escherichia coli by adsorbents and YA YHA, which were prepared by thermal activation of Moroccan oil shale Timahdit are very successful and open windows into other applications in environmental fields. In perspective, we plan to improve the textural and structural properties of adsorbents by searching the best conditions for activation and expansion of application fields on real samples.

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