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Synthesis and application of Algerian natural kaolins modified 13X zeolite for the treatment of real textile effluent

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ABSTRACT : In this study, 13X zeolite was successfully synthesized from natural local kaolins DD1, DD2 and DD3 via alkali fusion followed by hydrothermal treatment. Fusion with NaOH, followed by hydrothermal reaction, kaolinite and halloysite in kaolin sample were converted into 13X zeolite. The Three kaolins DD1, DD2 and DD3 issued zeolite 13X were characterized using X-ray diffraction (XRD) and scanning electronic microscopy (MEB). The adsorption experiments were done using the real textile effluent. The results show that the adsorption is efficient and feasible at ambient temperature on DD1 kaolin issued 13X compared to 13X zeolite synthesised by DD2 and DD3 with good discoloration efficiency. Experiments with real textile wastewater showed an improvement of physico-chemical characteristics (COD, MIS, PO₄³⁻, NO₂⁻) compared to the untreated effluent.

Key words : Zeolite 13X, Kaolins, Adsorption , Real textile effluent.

1. Introduction

Textile effluents are among the most polluting industrial wastes, and the problem of treatment and disposal of such wastes requires much attention. A certain amount of dye is lost in the manufacturing processes and remains in their effluents. Moreover, it may be released in aqueous effluents such as in river streams which become colored. Moreover, textile dyes are toxic to living organisms due to their low biodegradability and persistence in the environment, and their presence in wastewater induces a risk to human health [1, 2]. Therefore, effluents have to be treated carefully before discharge. The treatment capacity of existing industrial effluents represents about 20 million m³ per year, some 10% of the volume of wastewater generated.

The kaolin may be a good alternative low-cost adsorbent because it is the most abundant kaolinite-rich rock in Algeria. Indeed, Algeria possesses in the East, an estimated amount of millions of tons of clays that need to be promoted.

However, many works pointed out the adequacy of fly ash as feedstock in the syntheses of zeolites [3-5]. The synthesis of zeolites from Kaolin is well-known and

described by Barrer et al. [6]. Further raw materials such as high silica bauxites, halloysites, and montmorillonites are also used for the synthesis of zeolites [7]. Nevertheless, to the best of our knowledge, no previous effort has been taken to use natural local kaolins DD1, DD2 and DD3 as starting material to directly synthesize 13X zeolite.

The aim of the present work is to investigate the treatment of real textile effluent, onto Algerian kaolins from Djebel Debagh DD1, DD2 and DD3. The physicochemical characteristics were measured and compared in the three kaolins.

2. Materials and Methods

2.1 Materials

The local kaolins DD1, DD2 and DD3 were purchased from Djebel Debagh, the region of Guelma in the East of Algeria. The kaolins were prepared as a starting material for the present experiments after crushing and air-drying. DD1, DD2, DD3 kaolins were sampled in an active kaolin mine, formed by hydrothermal alteration processes in the region of Guelma (Djebel Debagh, Algeria) [8,9].

The real textile wastewaters effluents were supplied from the COTITEX (Factory specialize in the fabrication of military tissues Algeria). During the fabrication and dyeing processes two types of dyes (vat and dispersive) and different kinds of chemical substances were used such as peroxide, caustic soda, and auxiliaries.

2.2 Synthesis of 13X zeolite

13X zeolite sample was prepared using low-grade natural kaolin as silica–alumina source via alkali fusion followed by hydrothermal treatment. Sodium hydroxide and kaolin with the weight ratio of 2:1 were milled at 200 C for 4 h. The fused mixture was cooled and added to water. The slurry obtained was vigorously agitated for 2 h at 50°C for homogenization (450 r/min), and then was crystallized at 90 C for 8 h (300 r/min). Finally, the solid was separated by filtration, washed several times and dried.

2.3 Dynamic adsorption of textile effluent

Adsorption studies were performed in batch to assess the adsorptive capacities of real textile effluent. Adsorption experiments were carried out in 200 ml of effluent, containing 0.1 g of kaolins (DD1, DD2, DD3) and 13X zeolite synthesised from kaolin. The agitation is at a constant stirring speed of 450 rpm at natural pH (7.0) for 140 min. The samples were then centrifuged for 15 min at 5000 rpm, and the left out concentrations in the supernatant effluent were analyzed. The analysis of physicochemical parameters (pH, conductivity MIS, COD, NO₂⁻, PO₄³⁻, SO₄²⁻, NH₄⁺) were carried out.

The NO₂⁻, PO₄³⁻, SO₄²⁻, NH₄⁺ elements are dosed by the spectrophotometer Photolab Spektral brand WTW with dosing kits adapted for each element. COD is also measured by this method.

Suspended matter (MIS) represents all the mineral and organic particles contained in the waters and measured according to the reference cited in Rodier, 1996.

3. Results and discussion

The possibility to reduce COD, MIS and other elements using 13X zeolite synthesised from DD1, DD2 and DD3 local kaolins in the treatment of a real textile effluent were tested. Physicochemical analysis of the textile effluent before and after treatment over the 13X zeolite is summarised in Table 1.

The table shows a decrease in the quantities dosed for the treated effluent and it is more significant for the 13X zeolite synthesised from DD1 (13X zeolite/DD1) kaolin compared to other 13X zeolite synthesised from DD1 and DD2 kaolins.

On the other hand, the synthesized 13X zeolite/DD1 zeolite has a high efficiency by adsorption of the effluent then 13X zeolite/DD2 and 13X zeolite/DD3. The chemical and mineralogical composition of DD1 kaolin would increase the adsorption different elements presents in effluent (Figure 1).

Table 1. the physicochemical characteristics of the effluent before treatment and after treatment with local kaolins and 13X zeolite synthesised from kaolins

	Effluent before treatment	Effluent after coagulation/ flocculation	Effluent after adsorption on DD1	Effluent after adsorption on DD2	Effluent after adsorption on DD3	Effluent after adsorption on 13 X /DD1 zeolite	Effluent Afetr adsorption on 13X/DD2 zeolite	Effluent after adsorption on 13X/DD3 zeolite
pH	12	7.70	7.92	8.03	8.06	7.90	7.95	8.16
conductivity	1.8	1.5	1.4	1.5	1.4	1.5	1.5	1.1
DCO	1174	869	481	525	555	565	569	587
MES	119	20	3	9	4	0.1	1	0.5
SO₄²⁻	800	650	483	500	549	423	469	566
PO₄³⁻	1.52	1.13	0.83	0.97	1.08	0.75	0.88	1.07
NH₄⁺	0.31	0.18	0.12	0.13	0.15	0.10	0.11	0.14
NO₂⁻	0.51	0.33	0.16	0.23	0.28	0.15	0.20	0.21

4. Conclusion

The 13X zeolite was synthesized from low-grade natural local kaolins DD1, DD2 and DD3 via alkali fusion followed by hydrothermal treatment. The prepared 13X zeolite/DD1 exhibited adsorption capacities which exceed those of 13X zeolite/DD2 and 13X zeolite/DD3. The overall data show the high potential of the 13X zeolite synthesised from DD1 for the treatment of dyeing's waste water.



Figure1. Effluent before treatment and after treatment with 13X zeolite synthesised from DD1 kaolin

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