

# Mineralogical Quantitative, Crystalline and Morphological Characterization of Siliceous Material from the Rocks Modified by Ferric Oxide and Oxyhydroxide

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The main phases of ferric oxide in nature are in increasing order of crystallinity, ferrihydrite, maghemite ( $\gamma\text{-Fe}_2\text{O}_3$ ), lepidocrocite ( $(\gamma\text{-FeOOH})$ ), hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ) and goethite ( $\alpha\text{-FeOOH}$ ) (Schwertmann et al., 2000). Among these materials is diatomite ( $\text{SiO}_2 \cdot n\text{H}_2\text{O}$ ), also known as diatomaceous earth or kieselguhr, which refers to a soft light rock composed of amorphous silica microfossils of aquatic algae (Lemonas, 1997). The diatomite used in this study comes from the region of Sig (50 km from the city of Oran) in western Algeria, in the form of a white powder (Fig. 1).

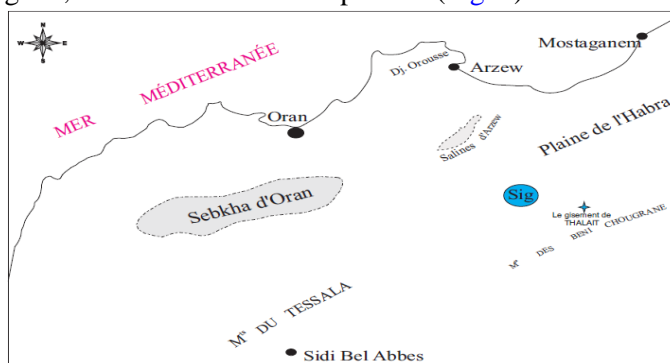


Fig. 1. Geographical location of layer of diatomite (Kieselguhr) in the area of Sig (west of Algeria)(Perrodon, 1952, 1957; Thomas, 1985; Mansour, 1991; Mansour et al., 1994)

Figure 2 shows the raw diatomite DB in powder (Kieselguhr).



Fig. 2. The raw diatomite DB in powder (Kieselguhr) the area of Sig «the west of Algeria »

45g of raw diatomite of Sig DB were immersed in 300 mL of 6M NaOH at 90°C for 2 h to partially dissolve the Si (Al-Degs et al., 2001). The mixture was immediately added to 300 mL of 4M concentration  $\text{FeCl}_2 \cdot 4\text{H}_2\text{O}$  ferric chloride tetrahydrate solution and stirred and oxidized in room temperature air for 24 hours. The solid obtained by centrifugation was washed with distilled water and oxidized by air for 24 hours. The mixture was dried at 105 °C in an oven for 24 h. The product of this process were called DMF4 respectively (Figs. 3,4).



Fig. 3. Diatomite modified by iron (DMF4) during stirring Fig. 4. Iron-modified diatomite (DMF4) after agitation and oxidation by air

The mineralogical analysis of iron-modified diatomite "DMF4" shows the predominance of the three oxides which are: Iron (III) oxide ( $\text{Fe}_2\text{O}_3$ ) 53.161% ; sodium oxide ( $\text{Na}_2\text{O}$ ) 25.5% ; silica ( $\text{SiO}_2$ ) 12.942% and the presence of the oxides of low mass percentages which are: calcium oxide ( $\text{CaO}$ ) 1.19% ; alumina ( $\text{Al}_2\text{O}_3$ ) 0.566% ; magnesium oxide ( $\text{MgO}$ ) 0.198% ; potassium oxide ( $\text{K}_2\text{O}$ ) 0.349% ; 0.066%  $\text{TiO}_2$  and other 6.378%. The X-ray diffractogram of diatomite modified by ferrihydrite "DMF4" is given in Figure 5.

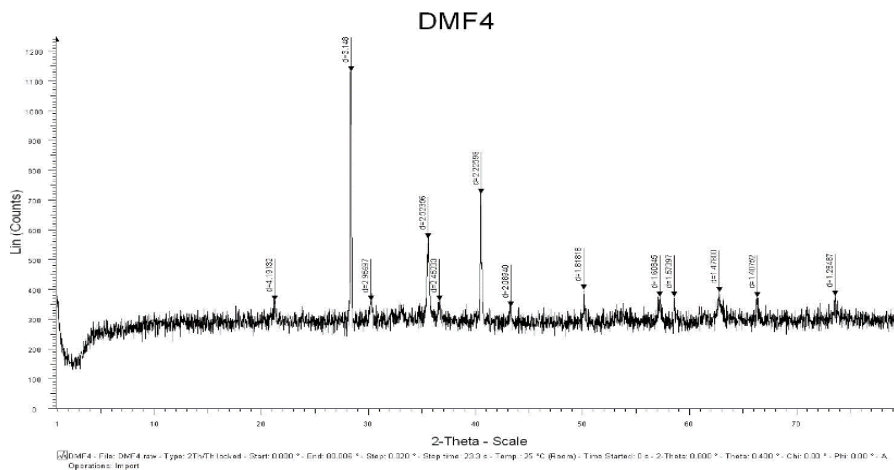


Fig. 5. X-ray diffractogram of iron-modified diatomite "DMF4"

Figure 5 shows the evolution of the intensity as a function of the scanning angle. For quartz ( $\text{SiO}_2$ ): the peaks at ( $2\theta=36.8^\circ-50.3^\circ-66.3^\circ-73.5^\circ$ ), with interarticular distances ( $d=2.45-1.81-1.40-1.28$ ) [1]. For hematite ( $\alpha\text{-Fe}_2\text{O}_3$ ): the peak at  $2\theta=35.5^\circ$  with the interarticular distance  $d=2.52$  [1]. For goethite ( $\alpha\text{-FeOOH}$ ): the peak at  $2\theta=21^\circ$  with the interarticular distance  $d=4.19$  [1] (ASTM file 17-536). For magnetite ( $\text{Fe}_3\text{O}_4$ ): peaks at ( $2\theta=43^\circ-57^\circ-62.5^\circ$ ) with interarticular distances ( $d=2.08-1.60-1.47$ ) [1]. For maghemite ( $\gamma\text{-Fe}_2\text{O}_3$ ): the peak at  $2\theta=43^\circ$  with the interarticular distance  $d=2.08$  [1]. Figure 6 shows scanning electron microscopic observation (SEM) of diatomite modified by ferrihydrite with calcination at  $600^\circ\text{C}$ .

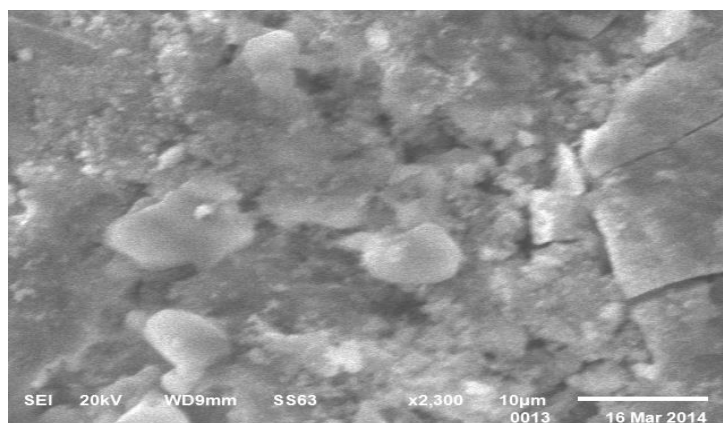


Fig. 6. SEM observation of ferrihydrite-modified diatomite with calcination at 600°C "DMF4" with magnification (x2300).

There are two main types of DMF4 model : The central particles of DMF4 have a diameter of approximately 1.6-8  $\mu\text{m}$  and a thickness of several microns. The pinnate particles of DMF4 have a length of ~3.3-15  $\mu\text{m}$  and a width of ~1.6-7.6  $\mu\text{m}$ .

**Keywords:** Diatomite, ferrihydrite, calcination, DMF4

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