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ORIGINAL ARTICLE

Synthesis and characterization of silica gel from siliceous sands of southern Tunisia

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Abstract The present work aimed to achieve valorization of Albian sands for the preparation of sodium silicates that are commonly used as a precursor to prepare silica gel. A siliceous sand sample was mixed with sodium carbonate and heated at a high temperature (1060 °C) to prepare sodium silicates. The sodium silicates were dissolved in distilled water to obtain high quality sodium silicate solution. Hydrochloric acid was then slowly added to the hydrated sodium silicates to obtain silica gel. The collected raw siliceous sands, as well as the prepared silica gels, were characterized by different techniques, such as X-ray fluorescence (XRF), X-ray diffraction (XRD), scanning electron microscopy (SEM) and thermal analysis (DSC). XRF confirmed that the detrital sand deposits of southern Tunisia contain high amounts of silica, with content ranging from 88.8% to 97.5%. The internal porosity varied between 17% and 22%, and the specific surface area was less than 5 m^2/g. After the treatment described above, it was observed that the porosity of the obtained silica gel reached 57% and the specific surface area exceeded 340 m^2/g. Nitrogen adsorption isotherms showed that the prepared silica gels are microporous and mesoporous materials with high adsorption capacities. These results suggest that the obtained silica gels are promising materials for numerous environmental applications.

1. Introduction

In southern Tunisia, the Dahar plateau extends from Tataouine in the north to the Tripolitain border in the south and is bordered to the east by the Jeffara coastal plain. The stratigraphic succession covers all Mesozoic periods and provides very important reserves of siliceous sands (Busson, 1967; Ben Ismail et al., 1989; Benton et al., 2000). These deposits, which are exposed along Dahar cliffs, are excavated for use in building. However, investments in these natural resources remain below the expected levels because of the lack of detailed studies

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encouraging developers to focus on the exploitation of silica sands.

For this reason, Tunisia is pursuing policies aimed at expanding the use of local materials, such as carbonates, clay and silica sands. Many works have been undertaken to deepen knowledge of facies, thickness variation and the physical and chemical characteristics of Albian sands of southern Tunisia (Bouaziz et al., 1989; Louhaichi, 1991; Bouaziz, 1995). High purity siliceous materials are used for glass making (Louhaichi, 1991) and, more recently, for various environmental applications (Besbes, 1999; Jesionowski, 2002; Marzouk et al., 2004).

Silica gels, which are known for their high specific surface area and their good gas adsorption capacities, could be produced from sodium silicate solutions by hydrothermal methods using sol-gel processes (Bouaziz et al., 1993; Marzouk et al., 2004).

This work aimed (1) to characterize a local material (silica sands), (2) to prepare a local adsorbent that would partially substitute for imported zeolite and active carbon and (3) to seek possible environmental uses for local silica sands.

2. Materials and experimental methods

2.1. Materials

For the purpose of this study, six sand samples were collected from the Albian continental formation outcropping in Douiret (S1), Oum diab (S2, S3), Ouni (S4) and Dehibat (S5, S6) in Tataouine district (southern Tunisia) (Fig. 1). The continental deposits of the Oum diab formation consist of 15 m thick sand layers alternating with shale. At the ''bled Oum diab'' location, outcropping beds contain up to 20 m of loose sands. Special attention was devoted to this location due to the promising physicochemical characteristics of the deposits.

2.2. Physical and chemical characterization

Prior to analyses, samples were dried for 24 h at 105 °C under vacuum conditions. Grain size distribution was carried out by dry sieving. The chemical composition of the studied samples was determined using X-ray fluorescence with an ARL® 9800 XP spectrometer (Thermo electron corp., Germany). Differential scanning calorimetry (DSC) was performed with a Perkin Elmer thermal analysis system (PerkinElmer Inc., Germany). About 5 mg of < 63 μm-sized samples were heated from room temperature to 600 °C at a heating rate of 10 °C/min under air atmosphere. The total pore volume and percentage of porosity were determined by pycnometry. X-ray diffraction patterns were obtained by an X-ray diffractometer (''PANalytical X' Pert High Score Plus'', The Netherlands) equipped with a dual goniometer of Cuα ($\lambda = 1.5406$ μm) using an acceleration voltage of 40 kV. The diffraction angle was scanned from 5° to 70° 2θ, at a range of 2°/min. SEM images were obtained using a Philips XL-30 electron microscope (Philips Electronics corp., The Netherlands).

The specific surface area and pore size distribution were determined with a SORPTOMATIC 1990 (CE instrument,

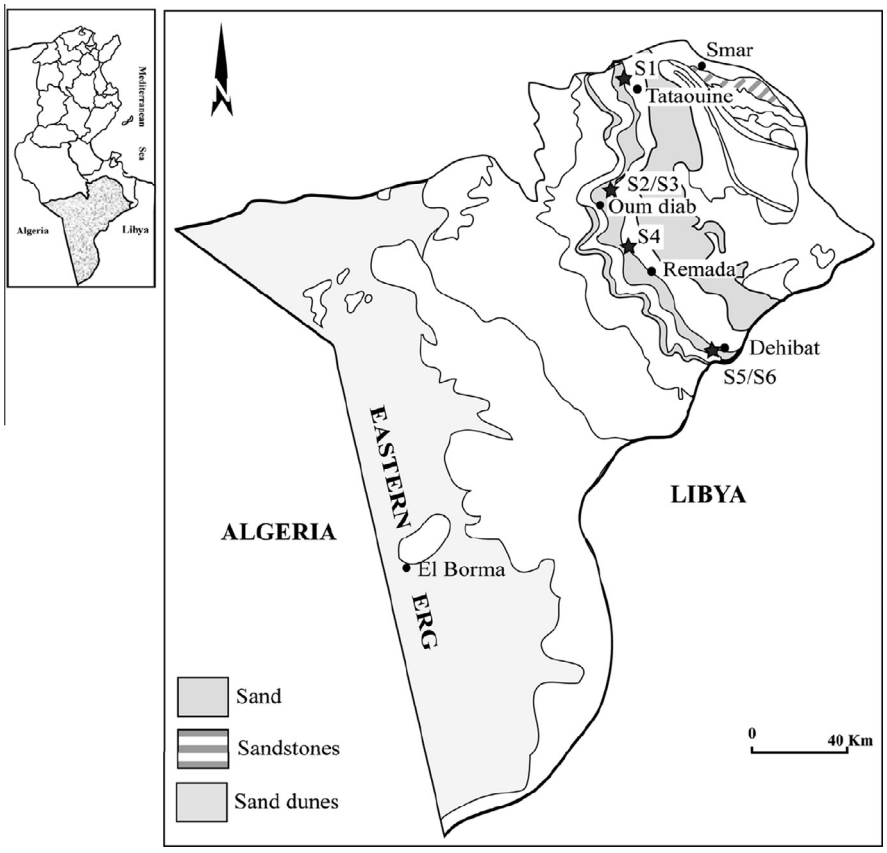

Figure 1 Outcrops of the continental deposits of southern Tunisia (modified after Bouaziz et al., 1989) and locations of the collected sand samples (stars).