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Characterization and separation of pyrite from Abu Tartur black shale

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ABSTRACT

This work aimed for pyrite separation from Abu Tartur black shale as a source of sulfur to be an added economic value of Abu Tartur area. The considered samples in the present work were collected from a core drilled in Abu Tartur plateau representing the pyrite-rich black shale of the U. Cretaceous age. Sample characterization was carried out using petrographic microscope, XRD, DTA/DTG, C/S and XRF techniques. Clay minerals, silt-sized quartz, calcite, and hematite were the main minerals associating pyrite (5.34%). Liberation behavior of the sample was about 80% below 5 μm .

Sample processing was achieved through one-day soaking followed by classification using 1 inch Mozley hydro-cyclone where about 35.5% by weight went to underflow and 64.5% went to overflow. The underflow product was subjected to an advanced gravity separation process using SB-40 Falcon Concentrator through a CCD statistical design prepared by Design-Expert 6.0 software proposed to optimize the separation process through a study for the effects of frequency (Hz) and water pressure (Psi) on both assay and recovery of the sulfur-rich heavy fraction.

A heavy concentrate weighed 10.90% with inorganic sulfur content reached 11.37% (21.24% pyrite) with overall recovery (50.01%) was obtained after two cleaning at the optimum conditions.

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1. Introduction

The Earth's climate has alternated between greenhouse (warm) and icehouse (cool) modes throughout the Phanerozoic. As the global warming has many environmental effects; global sea level raise, increasingly powerful hurricanes, as well as enhanced amounts of precipitation. The Mesozoic-early Cenozoic is known as a typical greenhouse period caused largely by increased CO₂ from elevated global igneous activity. The mid-Cretaceous is marked by a major warming peak [1]. Schlanger and Jenkyns [2] termed these widespread depositional black shale intervals "Oceanic Anoxic Events". These circumstances of weakly ventilated restricted basins under regional anoxic conditions formed a preferred media for organic-rich sediments accumulation between the Middle Jurassic and mid-Cretaceous. Large volumes of hydrocarbons were derived through the decomposition and maturation of an amount of these sediments under suitable conditions of pressure and temperature.

The potential reserves of liquid hydrocarbons derived from oil shale in the world were found to be greatly exceeding the known

reserves derived from petroleum. On the other hand, some of these sediments had no chance to be matured and preserved as kerogen-rich sediments known as black (oil) shale. Many of these deposits contain small, but ubiquitous amounts of sulfide minerals including pyrite and/or marcasite formed diagenetically by microbial processes usually evolve biogenic gases including hydrogen sulfide; this provides the euxinic conditions needed for pyrite formation. Also, redox-sensitive elements (e.g., uranium, vanadium, zinc, molybdenum, cadmium, chromium and selenium) are often enriched in these sediments. Many workers have reported data concerning enriched values of trace elements in oil shale throughout the world [3–6].

Black (oil) shales are widely distributed over the world with reserves equivalent to about 3.2 trillion barrels all over six global continents. In many countries shale oil have been obtained from these sediments through thermal (retorting) processing in many countries as Estonia, Russia, Australia, USA, Brazil, Germany, and Scotland. Till now, little exploitation was applied on these sediments due to the technical issues affecting mining and processing of black shale. Cost and environmental impacts are the main unfavorable issues. A high level of harmful atmospheric emissions – especially hydrogen sulfide – produced through processing stages and exceeds those of coal.

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Pyrite is considered to be the main mineral phase that causes the formation of large quantities of hydrogen sulfide during oil shale retorting. Also, it was found that at the retorting temperatures, pyrite reacts with the organic matter, generated from kerogen, robbing it of hydrogen, thus resulting in a poorer product and/or a lower yield of oils. So, many inventors tried to eliminate or reduce the amount of pyrite converting it into sulfur using acidic solutions as cerium IV salt solution or cautiously using mild oxidants as hydrogen peroxide to oxidize pyrites in the oil shale without affecting kerogen.

Black shales were discovered in Egypt in 1940s and were first undertaken in 1958 in the Soviet Union. It was found to be related to the giant phosphate belt in Duwi and Dakhla formations and aging Upper Cretaceous – Lower Tertiary. The in-place shale oil resources in Egypt were estimated by approximately 4.5 bbl in Quseir-Safaga area and 1.2 bbl in Abu Tartur area [7,8]. Recently, El-Kammar [6] reported that the “in-place geological reserves of oil shale of the 800 kcal/kg quality” in Quseir area is estimated to be more than 9 billion tons that can produce 5.48 BBLs equivalent upon retorting. Black shales in Egypt are now known for their interesting enrichments in various metals especially vanadium and nickel [9–12]. Moreover, the speciation of the heavy metals and uranium in the Egyptian oil shale is not yet studied. Pyrite was detected in many of mineralogical studies applied on many of these deposits; in Dakhla Formation west Quseir area [13], in El-Nakheil oil shale in Quseir area [14,15], and in Duwi black shale in Abu Tartur phosphate mine and reached about 6% [16,17].

The oldest sedimentary rock in Abu Tartur plateau is the cross-bedded non-fossiliferous sandstone of the Nubia formation (Cenomanian to pre-Campanian). It is conformably overlain by the variegated shales of the Quseir formation which is overlain by the Duwi (phosphorite) formation of late Campanian to early Maastrichtian in age [18]. The Duwi formation consists lithologically of phosphate beds interbedded with black and gray shale, sandstone, siltstone and glauconite beds. The Dakhla formation overlies the Duwi formation and is mainly composed of black and dark gray shale or marlstone with exogyra limestone at the middle and it is capped by the Paleocene Kurkur (limestone) formation.

This work demonstrate an unprecedented study on black shale of the upper Duwi and Middle Dakhla formations in Abu Tartur plateau subjected to a two-fold study aiming for investigating their mineralogical characteristics focusing on pyrite to determine its amenability to be separated as an additional economic value to black (oil) shale of Abu Tartur area. Besides, pyrite separation will be a useful tool in reducing the harmful hydrogen sulfide emissions associated to oil shale retorting and this subsequently reduces the technological and environmental hazards of the future oil shale mining and processing in Egypt.

2. Materials and methods

2.1. Material

The black shale sample considered in the present work (7.22 m thick) was collected from borehole drilled by Egyptian Mineral Resources Authority and sponsored by Dana Gas[®] Egypt as a part of the Oil Shale project (2006–2010). The borehole was drilled in the Maghrabi-Liffiya sector due southeast of Abu Tartur plateau. The collected sample covered depths between 162 and 163.4 m from the upper part of the Campanian Duwi formation (1.4 m thick) and between 103 and 110 m, where a 25 cm of dolosparite sample lies between 109 and 110 m was excluded-covering the middle part of the Maastrichtian Dakhla formation (6.75 m thick). The bulk sample was crushed to pass through a 22 mm screen and subjected to characterization and processing.

2.2. Sample characterization

2.2.1. Thin and polished sections preparation

Samples for thin and polished sections were picked for petrographic analysis systematically as one sample for each 1/2 m or for every change in lithologic macrofacies or textural variation. Sections were prepared and examined under the petrographic microscope for composition and texture. To complete sample characterization XRD, DTA, XRF, C/S analyses were performed. The final pyrite concentrate was characterized and evaluated using XRD, C/S, and XRF techniques.

2.2.2. Mineralogical and chemical analyses

A BRUKER X-Ray Diffractometer (Germany) of type AXS D8 ADVANCE with Cu target ($\lambda = 1.540 \text{ \AA}$ and $n = 1$) at 40 kV potential and 40 mA current was used for characterization and minerals identification. The powdered samples were analyzed using XRD with scanning speed of $2^\circ/\text{min}$. Types of the phases in the samples were identified using the X-ray powder data file, published by the American standard for testing material (ASTM).

To separate the clay fractions, 10 g of the sample was transferred to 600 ml beakers and treated with dilute 10% hydrochloric acid with slight heating to remove carbonates. The residue was washed with distilled water and then treated with 30% H_2O_2 to remove organic matter. After complete disaggregated, the sample was washed with distilled water several times until completely suspended. The suspended clay fraction ($<2 \mu\text{m}$) was mounted on glass slides by dropper and left to dry in air. Three oriented slides were measured; one was untreated (oriented), the second was saturated with ethylene glycol vapor at 60°C for 1 h, and the third was heated at 620°C for 2 h.

The thermal behavior of the sample was recorded from thermo-gravimetric (TG/DTG) and differential scanning calorimetric aspects using NETSCH STA 409 C/CD instrument. TG and DSC signals were recorded under heating conditions up to 1000°C with heating rate of $10^\circ\text{C}/\text{min}$. Heating took place in DSC/TG pan made of aluminum oxide.

An Axios, Sequential WD-2005 device was used for determination of the chemical composition. The elemental oxide composition of the sample was determined based on dry conditions. Sulfur percent was determined for the powder sample using Eltra 2000 carbon sulfur analyzer through combustion and measurement of the combustion gases by means of infrared absorption. During combustion, the sulfur components present in the sample are oxidized to form SO_2 . The usual combustion temperatures are $1250\text{--}1400^\circ\text{C}$. The signals emitted from the infrared cells are selective and correspond to the SO_2 concentrations in the gas mixture. They were electronically linearized and integrated, divided by the sample weight and digitally displayed as S%. The standard gravimetric analytical method [19] was used to follow up sulfur concentration during the different processing stages.

2.2.3. Size analysis

Wet and dry sieve analyses were employed by passing a known weight of the sample through a prepared series of sieves having different sizes from coarser to finer sieves in a successive way. The remaining amount of retained material over each sieve collected, weighed, and the percent of each size fraction was calculated. The passed cumulative weight percents were graphically represented with a logarithmic scale on X-axis.

2.3. Processing and equipment

2.3.1. Sample preparation for processing

Shale as a rock is characterized by its fissility and/or lamination [20]. This property of shale and also the presence of

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