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Evaluation of Egyptian diatomite for filter aid applications

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Abstract. Diatomite is fine-grained fossil siliceous sedimentary rock. It consists of micronized amorphous silica with a special porous structure. In this study, a technological diatomite sample was subjected to characterization by XRD, XRF, SEM and TEM techniques. Results showed the high grade of the sample with silica content exceeding 90% SiO2. The main accompanying minerals were fine sands, calcite, and kaolinite. The sample was subjected to gentle size reduction to avoid ore cell destruction. The attrition +74µm fraction was rejected, where the -74µm slurry continued refining via the 7.62cm Sprout Bauer and the 5.08cm Mosley hydrocyclones. The -25µm fraction was investigated as the final refined diatomite product.

keywords: diatomaceous earth, diatomite, porous materials, surface texture, filtration, processing

1. Introduction

Diatomite ores are microscopic single-celled algae with characteristic rigid cell walls (frustules) composed of amorphous silica (Fig. 1). They consist normally of 87-91% silicon oxide (SiO_2), with significant quantities of alumina (Al_2O_3) and iron oxide (Fe_2O_3). The physical properties of natural and processed diatomite that provide unique commercial value in a broad spectrum of market end-uses include ornate fine structure, low bulk density, and high porosity and surface area. The properties of equal importance are mild abrasiveness, high absorptive capacity, insulating ability, relative inertness, and high brightness. End-use markets are diverse and range from insulating brick and absorbents through quality sensitive filter aids and premium quality functional fillers (Sterrenburg and Seckbach, 2007).

Although many thousands of species of diatoms have been classified, Figure 2, diatoms of salt water origin have been generally preferred as a source of filter aid materials because the major contaminant in saltwater diatom deposits tends to be sand or grit which is easily removed. In contrast, the major contaminant in fresh water deposits tends to be clay which has been difficult and costly to remove. In addition it has been considered that a large variety of particle shapes of marine diatoms gives the most efficient filter medium, Figure 2. It has been discovered that many deposits of diatoms of freshwater origin are comprised substantially of the species Melosora Granulata which, when cleaned of clay impurities, provides a filter aid with superior filtration qualities (Potter, 2000; Sterrenburg and Seckbach, 2007).

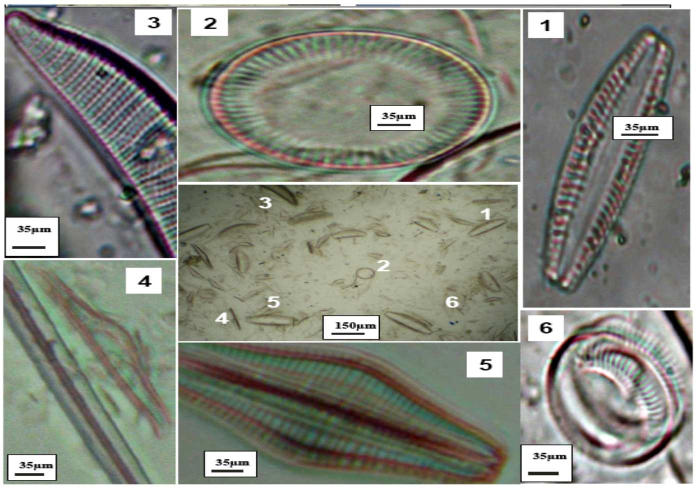


Fig. 1. Different shapes of diatomite particles

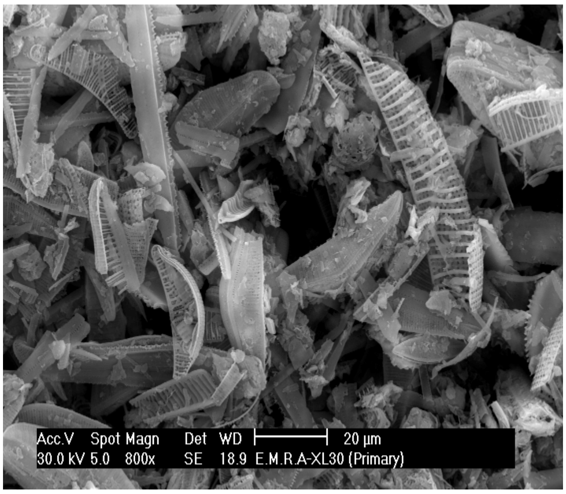


Fig. 2. Tremendous types of diatoms: recent and fossil, freshwater and marine

Diatomite is used in cleaning drinking water or industrial and sewage waste water. Sometimes it is blended with aluminium chloride or ferric chloride to enhance the filtration processes. From one to five percent, by volume, of polyacrylamide is preferably added to the mixture for use in sewage waste water treatment applications (Gordon et al., 2005).

Diatomaceous filter aids may be broadly categorized as falling into one of three grades - "natural", "calcined" and "flux-calcined" (Agdi et al., 1999; Alvarez et al., 1999; Andrews, 1991; Aytas et al., 1999; Engh, 1993; Harben, 1995; Skillen, 1995). The production of diatomaceous filter aid from diatomaceous ore is termed beneficiation. Natural diatomite is usually prepared by crushing the raw ore in a hammer mill and then drying the crushed material to remove substantially all moisture. The crushed material is then classified in air cyclones to remove the sand contamination and to effect a rough particle size separation. This initial processing causes undesirable breakage of frustules during the milling and classification steps. It was suggested that ultrasonic energy was an efficient means to induce a shear force to liberate the clayey particulates, which have particle size of -2µm, from the diatomaceous particles which were found naturally between 30-2µm, before classification through hydrocyclone classifiers (Agdi et al., 1999; Alvarez et al., 1999; Al-Wakeel, 2009; Andrews, 1991; Aytas et al., 1999; Engh, 1993; Neesse et al., 2004).

Diatomite may be used in a filtering process as a "pre-coat" or as a "body feed" depending on the nature of the material to be filtered. The shape and size of the filter aid particles is a relevant consideration for optimized performance. Particulate materials having a wide particle size distribution make efficient filter aids in terms of the clarity of filtrate, but their tendency to pack tightly results in very low filtration rates (Agdi et al., 1999; Alvarez et al., 1999; Andrews, 1991; Aytas et al., 1999; Crossley, 2000; Engh, 1993).

Coarse particles having a regular particle shape permit high filtration rates but will allow fine impurities to remain in the filtrate. Diatomaceous earths often comprise a mixture of substantially intact frustules together with a proportion of broken frustules (Christensen et al., 2001; El-Shafey et al., 2004; Lemonas, 1997; Loukina et al., 1994; Ridha et al., 1998; Shwabken and Tutinji, 2003).

Although many thousands of species of diatoms have been classified, diatoms of salt water origin have been generally preferred as a source of filter aid materials because the major contaminant in saltwater diatom deposits tends to be sand or grit which is easily removed. In contrast, the major contaminant in fresh water deposits tends to be clay which has been difficult and costly to remove. In addition it has been considered that the large variety of particle shapes of marine diatoms gives the most efficient filter medium (Breese, 1994; Carr, 1994; El-Shafey et al., 2004; Lemonas, 1997; Loukina et al., 1994; Martinovic et al., 2006; Ridha et al., 1998; Schuller, 1991; Shwabken and Tutinji, 2003).

Egypt is blessed with huge reserves of diatomite deposits, varying in grade from high, moderate to low rank (Abdel Aleem, 1958a; Abdel Aleem, 1958b; Basta et al., 1971; Basta et al., 1972; Faris and Girgis, 1969a, Faris and Girgis, 1969b, 1969; Hassan et al, 1999; Ibrahim, 2007; Said, 1962; Said, 1972; Zalat, 2002). This study focuses on the evaluation and processing of Gebel Elow El Masakheet Diatomite for filter aid applications. It is important to mention that the diatomite ore deposit; under investigation; covers a large area in the southwest of the El- Fayoum Depression in the area between Wadi El Rayan to the south east of Gabel Elow El Masakheet locality, Figure 3. This area is located to the south west of El- Fayoum city by about 28km. Figure 4. Illustrates the Landsat of these reserves that extend for bout 10km in the ENE-WSW direction and for about 5km in the NNW-SSE direction.

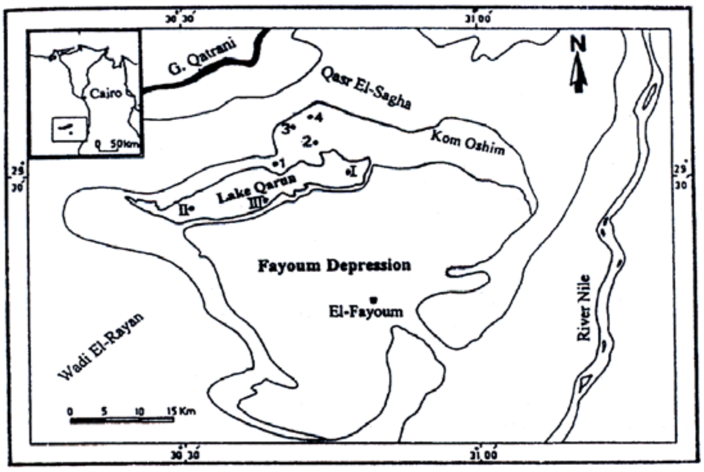


Fig. 3. Different diatomite deposits in El-Fayoum Depression, Egypt

2. Experimental

A representative sample from the well-exposed outcrop of the Gebel Elow El Masakheet diatomite was collected. The sample was gently crushed by hammers to -10cm. The primary crushed sample was left soaked in water over night and then transferred to the 40 liter capacity Denver attrition scrubber at 40-50 solid% and for 30min. The attrition process was in closed circuit with a 74µm Russell vibrating screen. The over screen product was collected as rejected fraction and subjected to evaluation and storage. The under screen slurry pulp was subjected to the 7.08cm Sprout Bauer and the 5.08 Mosley hydrocyclones after dilution to 20-25 solid %. The pressures inside the hydrocyclones were kept at maximum values reaching 345-379 kPa (50-55 psi), and 275 kPa (40 psi) for the two hydrocyclones, respectively. The products after hydrocyclone classification were evaluated with respect to diatomite wt. % and grade i.e. recovery (Schulz, 1970). The 5.08cm Mosley hydrocyclone overflow product was collected as the diatomite concentrate and directed to complete characterization. Whiteness measure of diatomite was measured (TAPPI, 1973). The concentrate product skeleton has been microscopically viewed using scanning electron microscope (SEM) of the type JEM-1230, JEOL and transmission electron microscope JSM T-20, JEOL. Evaluation of the concentrate product as filter aid was conducted via the microstructure analysis to provide reliable information about shape, structure, pore spaces and size.

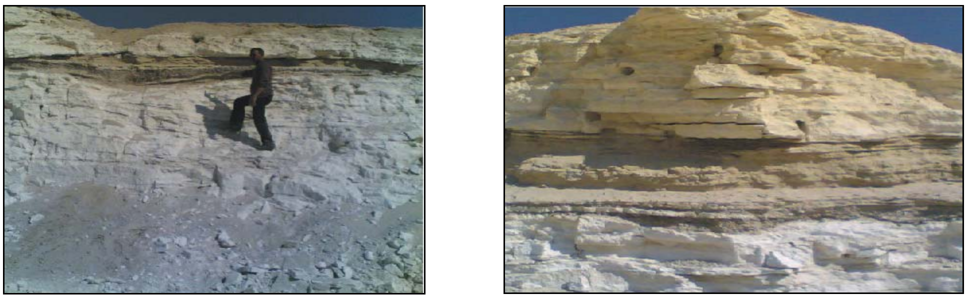
3. Results and discussion

3.1. Deposit field relations and stratigraphy

It is believed that the Gebel Elow El Masakheet deposit is a depositional basin, which was filled with lacustrine facies during the Holocene, as most places of El-Fayoum Depression. These lacustrine acies were characterized by diatomite-rich horizons intercalated with calcite matrix, sand and silt. These sediments rested unconformably on the Middle Eocene rocks. Stratigraphy and structural relationships between these sediments and surrounding rocks are less complicated and were affected by varied local depositional topography (Basta, 1971).

In general, the Gebel Elow El Masakheet diatomite deposit is of white to grayish white color. Sometimes these horizons are intercalating with each other, displaying textural lamination and fissures. They range in thickness between centimeters to meters, Figures 4a and 4b. Thickness of the deposits depends mainly on the geomorphology of the basin during their deposition (Basta, 1971]) The diatomaceous earth deposits occurred as jointed layers with gradation in colour from bright white at the base to pale white in the top, Figure 4a or as successive cycles of deposition separated from each other by beds of grayish white or yellowish white friable fine sand, Figure 4b It is important to mention here that diatomite deposits of type (a) were the only type under investigation in this study.

a



b

Fig. 4. a) the jointed fissile diatomite with gradation in colour from bright white at the base to off white in the top; b) white diatomite overlain by yellowish white blown sand enclosed with conglomeritic lenses of yellowish friable sand

3.2. Sample characterization

The microscopic studies and the XRD analysis of the Gebel Elow El Masakheet diatomite original sample showed that the sample was high grade diatomite composed of diatomaceous skeletons (frustules) containing minor amounts of calcite, kaolin, and crystalline silica minerals. The XRD pattern of the sample depicted clearly the highly crystalline peaks of calcite, clays, quartz, beside the amorphous peak related to the diatomite phase. On the other hand, Table 1 illustrates the chemical analysis of the original sample. The broad XRD peak between 2θ 9-19, as well as the high silica content of the sample showed the high grade of the diatomite sample. Table 2 depicts the density values of different minerals which constituted the diatomite ore sample under study, i.e. diatomite, silica, calcite and kaolinite.

Table 1. Chemical analysis of Gebel Elow El Masakheet diatomite original sample

Constituent	Wt.%
SiO_2	90.03
CaO	5.01
Al_2O_3	1.88
Fe_2O_3	0.08
LOI	2.20

Table 2. Specific gravity values of different minerals constituents of the diatomite ore sample

Mineral	Density, g/cm^3
diatomite	1.66
kaolinite	2.60
silica sand	2.65
calcite	2.71
iron oxide	5.10

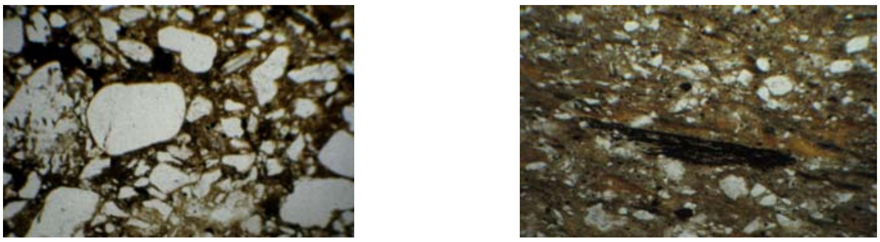
3.3. Processing of the crushed diatomite sample

Table 3 illustrates the refining processing stages of the sample. It was obvious that the attrition scrubbing-closed circuit with 74 µm sieving of the hammer crushed sample was efficient to liberate the diatomite particles from the attached fine silica (+74 µm tailings). Different separation tests using the 7.62 cm Sprout Bauer cyclone showed that at pressure rate of 345-379 kPa (50-55 psi), a tail fraction weighing 7.40% containing most of the calcite impurities and fine silica remains was separated, Figure 5. Separation results of the 5.08cm Mosley hydrocyclone indicated that, the diatomite separation is highly sensitive to changes in hydrocyclone pressure (Neesse, et al., 2004; Schulz, 1970; Svarovsky, 2000). At relatively low pressures, there was poor separation, where the diatomite was distributed indiscriminately between the overflow and the underflow cuts. With increasing pressure, the separation was improved till reaches its maximum separation efficiency >80% with high grade product (96.03% SiO_2) at 275 kPa (40 psi). It was noticed that by increasing the pressure sharp deterioration in separation of diatomite with respect to weight% took place. The measured mean diatomite particle diameter (d_{50}) was about 13 µm where d_{10} and d_{90} was 4µm and 26 µm, respectively (Fig. 6).

The physical characteristics of the final product are given in Table 4. From the data shown in Table 4, it could be concluded that the processed diatomite is amenable for filter aid application according to the international criteria (Carr, 1994 and Crossley, 2000). The morphology as well as the porous structure and the surface texture of the product sample are shown in Figs 9 and 10. SEM pictures show that the main diatomite structures were of types Cocconeis klamathensis and Cymbella prostrate, which related to fresh water environment, Figure 7 (Sterrenburg and Seckbach, 2007). The final product showed to have a pore diameter of about 0.25µm (Fig. 8).

Table 3. Refining results for the Gebel Elow El Masakheet diatomite original sample

Process	Wt.%	SiO_2%	Al_2O_3%	CaO%	LOI%
Original sample	100.0	90.91	1.88	5.01	2.20
Attrition scrubbing:					
Over Screen, +74µm fraction	9.31	96.44	0.18	3.07	0.31
Hydrocyclone classification:					
7.62cm Hydrocyclone UF Fraction, -74+45µm	7.40	31.30	3.70	36.20	28.80
5.08cm Hydrocyclone UF Fraction, +45+25µm	3.09	54.88	27.23	7.76	10.13
5.08cm Hydrocyclone OF Product, -25µm	80.20	96.03	0.91	1.65	1.41



a b

Fig. 5. a) Rejected +74µm fraction (silica and fossil remains) and b) rejected +45µm fractions (muddy calcite and fine silica) after the refining process

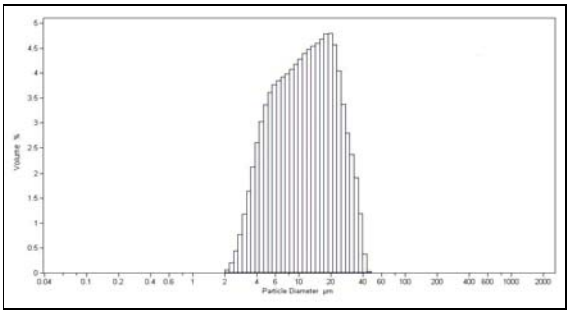


Fig. 6. Size distribution of the final diatomite concentrate

Table 4. Main physical characteristics of diatomite final product

Physical Properties	Final Product
Specific surface area, m^2 / g	195
Specific gravity, g/cm^3	1.66(crude) , 0.43 (dry base)
Water Absorption (% by weight)	262, 322.36 (After drying)
Oil Absorption (g /100 g)	139-208, 221.92(After drying)
Whiteness	97.70
% Passing 45 µm	100.00
Porosity %	51.49
Refractive index	1.462-1.558
pH	5.85

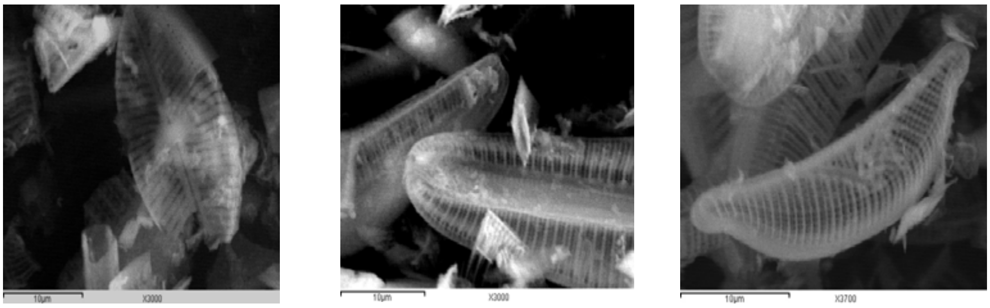


Fig. 7. SEM pictures of diatomite end-product

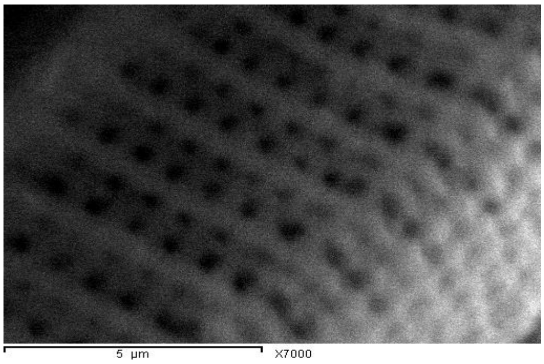


Fig. 8. The porous structure of diatomite particle end-product

4. Conclusions

In this study, a technological diatomite sample was collected from the Gabel Elow El- Masakheet locality, south west El- Fayoum Governorate, Egypt. The sample was subjected to characterization by the XRD and the XRF. The morphology as well as the porous structure and the surface texture of the product sample were observed using SEM and TEM techniques. The sample was subjected to gentle size reduction to avoid ore cell destruction. This was carried out through hammers crushing of the ore lumps, followed by attrition scrubbing of the -10cm crushed sample. Russell 74µm vibrating screening processed the attrition slurry to reject the overscreen organic and the earthy remains fraction, where the underscreen slurry continued to the refining circuit via the 7.62cm and the 5.08cm hydrocyclone units. The -25 µm product was collected, dried, and investigated as the final purified diatomite product. The final refined product recovery was 80.20% by weight and silica assay reached 96.03%. The D_{50} and D_{90} for the product were 13µm and 26µm, respectively. The morphology as well as the porous structure and the surface texture of the product sample were observed, showing that the pore diameter averaged 250nm. The study recommends that the refined diatomite of the Gabel Elow El- Masakheet locality, south west El-Fayoum Governorate, Egypt, is suitable for filter aid applications.

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