International Journal of Mineral Processing 94 (2010) 129–134

Contents lists available at ScienceDirect

International Journal of Mineral Processing

journal homepage: www.elsevier.com/locate/ijminpro

Improvement in filtration characteristics of diatomite by calcination

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ARTICLE INFO

Article history:
Received 9 March 2009
Received in revised form 8 January 2010
Accepted 7 February 2010
Available online 11 February 2010

Keywords:
Diatomite
Calcination of diatomite
Filtration
Filter aids
Beer filtration

ABSTRACT

This paper examines the calcination and filtration characteristics of diatomite. For this purpose, diatomite ore was calcined at 1000 °C in order to improve the material characteristics for use in filtration. The physical, chemical, thermal and micro-structural features of the raw and the calcined diatomites were then determined to compare them with those of the commercial filter aids currently used. In order to determine filtration efficiency of the diatomite samples, several filtration tests were carried out together with the beer samples and the commercial filter aids taken from a leading beer factory in Turkey. It is shown that the calcined diatomite could successfully be used for beer filtration after suitable arrangement of the particle size distribution, such that the highest possible flow rate and filtrate clarity are obtained.

1. Introduction

Diatomite is a non-metallic raw material composed of the skeletal remains of single-cell water plants and it has many useful features for use in several industrial applications such as high porosity and bulk volume, a porous and permeable structure, chemical resistance, high purity, large specific surface area, high adsorption capacity and good adsorptive properties (Hadjar et al., 2007; Khraisheh et al., 2004; Yuan et al., 2004; Yang et al., 2002; Al-Degs et al., 2001). Common industrial uses for diatomite include filtration of sugar syrup, beer, whiskey, wine, fruit juice, water, mineral or vegetable oils and pharmaceuticals (Advanced Minerals, 2005; Sulpizio, 1999).

After comminution, diatomite and its impurities are typically separated by use of air separators, mechanical mixing and cyclones or magnetic separators (Al-Wakeel 2009; Bentli, 2002; Mete, 1988). However, diatomite is usually treated by calcination or flux calcination methods to obtain better diatomite properties for filtration (Breese, 1994). Aluminum oxide, mostly present in the clay fraction, can clog the voids of diatomite particles and hinder its filtration properties. Iron oxide and organic materials are also undesirable impurities, especially in the food industry since they can change the color and organoleptical properties of the final product. Small particle sizes can result in filtration beds with low porosity and low filtration efficiency (Franca et al., 2003).

Firing of natural diatomite in a rotary kiln at 870–1100 °C (depending on the properties of the raw material and the method of production) is called calcination. In white (flux) calcination, 3–8% soda ash (Na_2CO_3) or salt (NaCl or KCl) is added to diatomite before the sintering or calcination stage. The flux allows iron oxides to enter a glassy phase where it is colorless and produces greater agglomeration of the diatom fragments. Variations in the kiln temperature, the amount and composition of the flux, and heating time in the kiln enable suppliers to produce products with different filtration rates (Martinovic et al., 2006; Kouteren, 1994). The product obtained after firing is cooled, ground and enriched by air separators and classified by size. Vasconcelos et al. (1998) has proved that the desired porous structure of diatomite could be obtained upon calcination. The advantages of calcination are removal of organic matter, removal of carbonate compounds, higher filtration rate depending on opening up of the diatom frustules, and aggregation owing to sintering and shrinkage of the particles.

The most important properties required by the industry are chemical composition, particle size, particle size distribution and micro-structure, dry and wet densities, water absorption capacity, pH value, color and specific and relative humidities (Advanced Minerals, 2002).

The purpose of this research is to investigate the possible use of Alayunt–Kutahya diatomite in various industrial applications including filtration by improving its features. Diatomite in this region was deposited in the Upper Miocene–Pliocene freshwater lacustrine basin (Gürel and Yıldız, 2007) through the effect of volcano-sedimentary processes which produced SiO_2-rich solutions. These solutions were mobilized by diatoms to construct their skeletons. In the area, volcano-sedimentary hosted freshwater diatomite deposition has horizontal layers. The reserve of diatomite in the area was calculated as approximately 15 million tonnes (Nuhoğlu and Elmas, 1999) with a SiO_2

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doi:10.1016/j.minpro.2010.02.004

content of 50–90% and is not commercially used at present. Therefore, creating an economic value out of Alayunt–Kutahya diatomite is of great importance for the region. For this purpose, physical, chemical, thermal and micro-structural properties of both raw and calcined ores were determined. The filtration characteristics of the ore were investigated using unfiltered beer samples. The results were then compared with those of the commercial filter aids taken from a leading beer factory in Turkey.

2. Material and method

2.1. Material

The ore used in this investigation was collected from Alayunt–Kutahya region of Turkey. The ore was used as both raw and calcined forms. Several tests were carried out in order to determine the properties of the materials used. For this purpose, chemical analysis of the raw and the calcined ores was made using a Spectra X-Lab 2000 brand XRF instrument. The results are given in Table 1.

The raw diatomite comminuted below 420 μm was calcined to obtain the calcined diatomite. In order to determine the physical features of the raw and the calcined diatomites, density, pH, color and permeability tests were also made. Density, pH and color measurements were performed using a Quantachrome-Ultrapycnometer 1000 brand picnometer, a Hanna-pH 211 microprocessor brand pH-meter and a Datacolor brand color measuring device, respectively. Permeability tests were made according to the standard coded as TS EN 12902 (2001). The results are given in Table 2. The particle size analysis was also conducted to determine the changes after the calcination process using a particle sizer, Malvern-Mastersizer 2000 (Fig. 1).

Phase analysis was performed to determine the mineral content of the raw and the calcined diatomite utilizing an XRD instrument (Rigaku Miniflex ZD13113) using X-rays of CuK_α ($\lambda = 1.54056$ Å) in 5–70° at a rate of 1°/min (Fig. 2). Finally, scanning electron microscopy (SEM) was used to determine diatom frustules using a Zeiss-Supra TM 50 VP brand SEM instrument (Fig. 3).

2.2. Method

2.2.1. Calcination process

Preliminary filtration tests on the raw diatomite could not reach the required permeability of the filter cake. Therefore, diatomite was calcined in order to improve its permeability and the other features (Bentli et al., 2004). For this purpose, thermal analysis (TG-DTA) was carried out in order to determine thermal behavior of the ore, i.e., the optimum calcination temperature and the weight losses expected during calcination. Thermal analysis was carried out using a Perkin-Elmer-Diamond model STA instrument and the results are given in Fig. 4. The test was conducted in a platinum crucible open to air. The instrument was programmed such that it provided heating from 25 °C to 1000 °C by 20 °C/min rate and remain at 1000 °C for 1 h and then a cooling from 1000 °C down to room temperature at the same rate. The thermal analysis revealed that 1000 °C is appropriate for calcination. The calcination process of the raw diatomite comminuted to −420 μm was accomplished in a Protherm-PLF 150/S model process controlled oven. The oven temperature was arranged such that it reached 1000 °C in 6 h and maintained this temperature for 1 h.

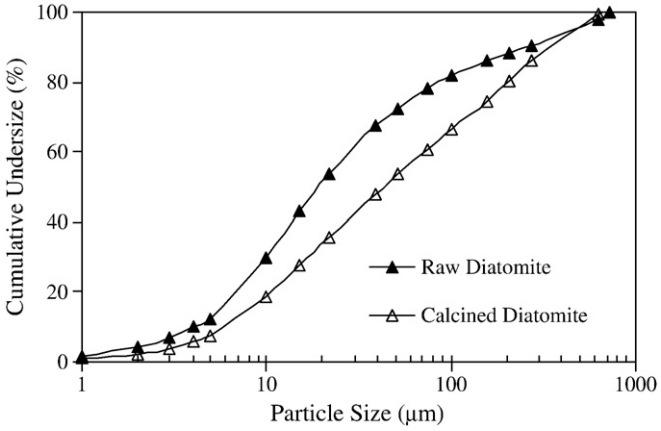

Fig. 1. Particle size distribution of the raw and the calcined diatomites.

Table 1
Chemical analysis of the raw and the calcined diatomites.

Constituents	Raw diatomite (%)	Calcined diatomite (%)
SiO_2	87.69	95.24
Al_2O_3	0.92	1.04
Fe_2O_3	0.28	0.35
Na_2O	0.64	0.30
K_2O	0.28	0.19
CaO	1.67	1.05
MgO	0.88	0.65
TiO_2	0.03	0.04
SO_3	0.11	0.09
L.O.I.	4.53	0.20
Total	97.03	99.15

Table 2
Physical properties of the raw and the calcined diatomites.

Measurements	Raw diatomite	Calcined diatomite
Density (g/cm^3)	2.29	2.34
pH	7.91	8.58
Color (L)	90.67	95.01
Permeability (m^2)	$2.15 \cdot 10^{-14}$	$4.74 \cdot 10^{-13}$

2.2.2. Filtration process

In these tests, the diatomite of Alayunt–Kutahya was compared with the two commercial filter aids (perlite and kieselghur) currently used in the beer factory. In order to make a reliable comparison, the particle size distribution was set similar to the commercial filter aids. Consequently, the upper particle size of the samples was chosen as 150 μm, similar to those of the commercial filter aids. The lower particle size of the samples, on the other hand, was chosen as 38 μm since the screen used at the bottom of the filtration unit had a size of 38 μm. However, this arrangement of particle size distribution could not achieve reasonable flow rates with the raw diatomite. Therefore, the subsequent tests were carried out with the calcined diatomite, commercially available kieselghur and perlite taken from the beer factory.

The results of particle size analysis of the materials used in the filtration tests (−150 μm +38 μm) are shown in Table 3. The results of density, pH, color, surface area (BET) measurements and permeability tests of these samples are summarized in Table 4. The specific surface area of the samples was determined by a Quantachroma-Nova 2200 apparatus using nitrogen as adsorbate (BET method).

In the filtration tests, the amount of filter aids was chosen as 40 g based on the size of the filtration unit. The volume of the beer to be filtered in each test, on the other hand, was chosen as 2500 ml and the turbidity of the unfiltered beer was measured as 145 NTU. Turbidity, pH and color measurements were made on the filtrate obtained after every 250 ml filtrate collected. Moreover, the amount of filtrate obtained after every minute was weighed and recorded. After the completion of each test, total filtration time and the height of the filter cake were also determined. Turbidity measurements of the beer samples were made using a turbidity instrument (Merk Türbiquant 1500 T) and color analysis was made with a Hellige color comparator.