Effect of Acid Treatment on the Physicochemical and Mineralogical Properties of Smectite Clays from Cameroon.

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INTRODUCTION

Acid activation is a useful method to modify the catalytic behavior, enhance clay properties, mainly their sorptive properties of clay minerals. Thus, acid treatment is commonly required when clays are used in industry, for example, as catalysts or catalyst supports in alkylation, dimerisation and polymerisation reactions and as a component in carbonless copying papers (Fahn and Fenderl, 1983). Probably the most widely used acid-activated clays are the bleaching earths (Anderson and Williams, 1962; Gates et al., 2002), which are capable of removing colour, odour and other impurities from cooking oils of vegetable and animal origin.

The focus of this study was to prepare clay materials by acid attack and determine the effect of acid treatment on the physicochemical and mineralogical properties of the new products obtained.

ACID ACTIVATION

Samples of 10 g were dispersed in 100 ml of 0.0, 0.5, 0.7, 1.0 and 4.0 N H₂SO₄ (prepared from H₂SO₄ 96% p.a. Fischer), the slurry was stirred for 2H at 80°C. At the end of each run, suspensions were filtered and the remaining solid phases were washed several times with pure water (MilliQ ultra filtration) to remove any unspent acid until the filtrates were neutral (test of 5% BaCl₂). The obtained activated clays were then dried at 110°C, gently ground in an agate mortar. The clay samples thus prepared are designed as BN-0.0, BN-0.5, BN-0.7, BN-1.0 and BN-4.0; S-0.0, S-0.5, S-0.7, S-1.0 and S-4.0 with the suffix indicating the concentration of H₂SO₄ used during the treatment. The acid treated samples were stored in the plastic bottles closed tightly for uses to the analytical methods.

ANALYTICAL METHODS

Structural modification

- X-ray diffraction (XRD)
- Fourier transform infrared spectroscopy (FTIR)
- Thermal analyses (DTA/TGA)
- Scanning electron microscopy (SEM)

Physicochemical properties

- Nitrogen adsorption – desorption isotherm (BET); for specific surface determination
- Cation exchange capacity (CEC)
- Cations dissolution
- Particle size distribution

RESULTS

1. Structural modification

2. Physicochemical properties of acid activated clays

- Particle size distribution:
  - Natural clay (1-10μm): 28.9%; Activated clay: 48.09% (sample BN)
  - Natural clay (10-20μm): 16.65%; Activated clay: 26.06% (sample BN)

- CEC: Cation exchange capacity decreases continuously with acid concentration
- Specific surface area (SBET): Specific surface area of acid-activated samples increased with the acid concentration, such that acid activation at 4 N gave rise to a specific surface area 134 m²/g and 84 m²/g respectively for BN and S clay samples, whereas in the untreated sample it was 65 m²/g and 74 m²/g

- Cations dissolution: Increase of H₂SO₄ concentration up to 4 N caused increase of cation dissolution (Ca, Mg, Na, Al, Fe and Si), and no amount of titanium is dissolved during acid treatment, indicating that anatase is not destroyed.

CONCLUSION

The results of this investigation show that the acid activation has caused remarkable change on the structure of smectite phase, the non-clay minerals like cristobalite, quartz and anatase remain unchanged during acid treatment. The specific surface area of activated clays increase, however the cation exchange capacity of clays decreases with increasing acid concentration.

References: