



## *Silica Impurities Removal on Bentonite Sample for Nanoclay Production*

H. Sedighi<sup>1</sup>, M. Irannajad<sup>1\*</sup> and M. Gharabaghi<sup>2</sup>

<sup>1</sup> Department of Mining and Metallurgical Eng., Amirkabir University of Technology, Tehran, Iran

<sup>2</sup> Department of Mining Eng., Tehran University, Tehran, Iran

(Received 10 April 2010; Accepted 16 October 2013)

### **ABSTRACT**

Removal of silica impurities on Reshm bentonite sample of Semnan province in Iran was investigated. Two processes of hydrocyclone and sedimentation treatment with sodium hexametaphosphate were used aiming for nanoclay production. Mineralogical studies indicated that the treated sample was a low grade Na-bentonite. The dominant impurities were cristobalite and quartz with low amounts of feldspar, gypsum, dolomite, calcite and zeolite. The Purification of the sample was processed by two methods of hydrocyclone and sedimentation treatment with sodium hexametaphosphate as depressant. The purified product was intercalated with octadecylamine. The evaluation of products was performed by XRD analysis. The purification results showed that hydrocyclone process failed to produce a purified montmorillonite product suitable for nanoclay application. Using sedimentation treatment with sodium hexametaphosphate as a depressant, a product with impurities of less than 5 percent, suitable for nanoclay application, can be produced. Intercalation of purified montmorillonite by octadecylamine showed that the application of sodium hexametaphosphate increase the basal spacing up to 36 °A.

### **KEYWORDS**

Montmorillonite, Nanoclay, Hydrocyclone, Sodium Hexametaphosphate, Intercalation, octadecylamine.

\* Corresponding Author, Email: iranajad@aut.ac.ir

## 1- INTRODUCTION

Bentonites are comprised predominantly of the smectite group of minerals. The most common are sodium and calcium montmorillonites. Nanoclays are ultra-fine clays usually considered to have a size of less than 5  $\mu\text{m}$ , commonly less than 2  $\mu\text{m}$ . These ultra-fine clays are very reactive and when incorporated into polymers, ceramics, inks, paints, and plastics, give some exceptional functional properties. Their properties are due to the large surface area to volume ratios. Performance improvements include increased tensile strength, heat deflection temperature, and flame retardance [1, 2]. Nanoclay should have less than about 5% by weight non-smectite impurities, preferably less than 2% by weight non-smectite impurities, including crystalline and amorphous impurities, in order to provide new and unexpected results in enhancement of polymer properties, when combined with the polymer in a nanocomposite composition [3].

Various methods were presented for bentonite purification; however two methods are well-known among them. In the first method, presented by Tributh & Lagaly, purification is based on chemical separation. In recent years, a fast method based on mechanical separation is presented by Amcol Inc. The Tribute & Lagaly method is based on separating various impurities by different processes. This method is designed to remove as many impurities as possible to achieve a pure clay mineral. However, the purified material may still contain impurities such as quartz. The Amcol method is based on passing the clay slurry through a series of hydrocyclones to remove the impurities, then centrifuging the clay to remove a majority of the particles having a size in the range of about 0.5  $\mu\text{m}$  to about 100  $\mu\text{m}$ . [2, 3]. Moreover, another method is presented by Paul Schick in 1973. In this method, crude bentonite is purified by pouring aqueous slurry of the clay into a very dilute aqueous solution of a technical grade of sodium hexametaphosphate, and allowing the mixture to stand for a few minutes, whereby almost the entire mineral impurities are precipitated [4]. This investigation focused on finding appropriate methods for the production of nanoclay in the Reshm bentonite ore deposit.

## 2- METHODOLOGY

The bentonite ore was beneficiated to improve its montmorillonite content by removing the associated impurity minerals, mainly cristobalite and quartz. The sample was primarily crushed using jaw and roll crushers to 100% under 2 mm in size. The crushed sample was transferred to the conditioning tank for preparation of a pulp of 5% solid. The pulp is transferred to the hydrocyclone for separation of the impurities at various operation conditions. In the sedimentation process, the slurry of 10% solid is diluted by a solution of sodium hexametaphosphate. The settled precipitate contains most

of the impurities, and the solids collected from the supernatant consist of almost pure montmorillonite. After each experiment, slurry and solid phases were separated by filtration and the remaining solid was dried at 60°C in an electrical oven. In this investigation, a number of experiments were carried out with varying operation conditions for the separation of silica impurities from the crude sample.

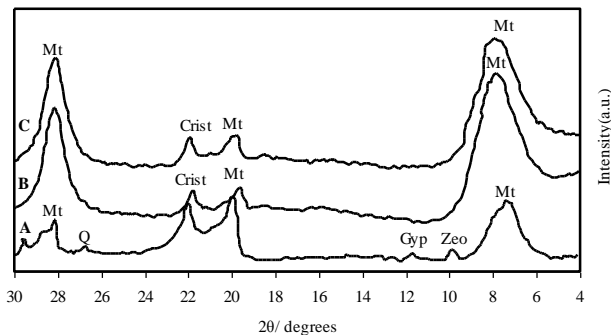
The Montmorillonite is intercalated with octadecylamine cations in aqueous solution by a cation exchange technique. Octadecylamine was protonated with HCl in-situ to obtain octadecylammonium cations [5, 6]. Water was used as reaction medium, and amine/clay and HCl/amine ratios were 2 and 1.5 respectively. Octadecylamine (ODA) was dissolved at 70°C in 150 ml water and then acidified with HCl (4cc). The purified Montmorillonite was first dispersed in distilled water at room temperature. The resulting suspension was then added to the amine solution and stirred vigorously for 1 h at 80 °C. The organically modified clays were recovered by filtration, washed with a large amount of water, filtered and then dried and evaluated by XRD analysis.

## 3- CONTRIBUTIONS

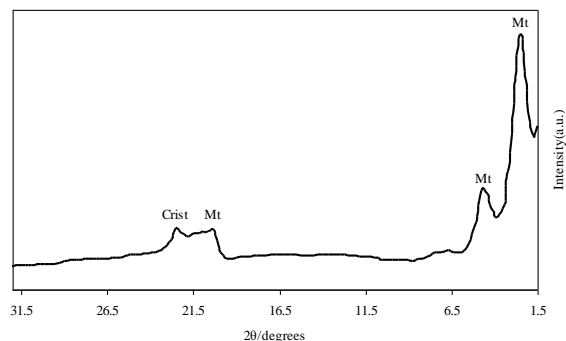
The results of beneficiation showed that applying a process based on sedimentation with sodium hexametaphosphate succeeded in removing much of the associated quartz, cristobalite and other impurities. The result of the modification experiments showed that the basal spacing of the intercalated montmorillonite increased to a size range of 22-36 Å°. This confirmed that the use of sodium hexametaphosphate has a positive effect on the process and improve intercalation efficiency.

## 4- SIMULATION RESULTS

A low grade Na montmorillonite beneficiation was investigated. The main associated gangue minerals were cristobalite and quartz with traces of other impurities. Montmorillonite has been purified with the sedimentation process, varying the sedimentation time and hexametaphosphate percentage in solution. The efficiency of purification depends on the sedimentation time, the best results being obtained in a range of 24 to 48 hours. In addition, a certain amount of the hexametaphosphate in the solution, influencing the process, is necessary to remove gangue minerals. The final concentrate has a minimum cristobalite content of 5%. The mineralogy, chemical analysis and physical properties of the final concentrate meet the specification required for application of the product in Nanoclay.



X-ray diffraction patterns for crude bentonite and purified bentonite (A: crude bentonite, B & C: purified bentonite)



X-ray diffraction patterns of purified sample after intercalation

## 5- REFERENCES

- [1] De Paiva, L.B., Morales, A.R., Valenzuela D., "Organoclays: Properties, preparation and applications", *Applied Clay Science*, 42, 8-24, 2008.
- [2] Clarey, M., Edwards, J., Tsipursky, S.J., Beall, G.W., Eisenhour, D.D., Amcol Intl, US Patent. 6050509, 2000.
- [3] Sedighi, H., Irannajad, M., Alinia, F., "Reshm bentonite beneficiation for nanoclay application", 2nd international congress on nanoscience & nanotechnology, 28-30 October, university of Tabriz, Tabriz, Iran, 2008.
- [4] Lars, A., "Cation exchange and adsorption on clays and clay minerals"; Dissertation, Submitted for the degree "Dr. rer. nat." of the faculty of mathematics and natural sciences, Christian-Albrechts Universität, 2003.
- [5] P. Schick, U.S. Patent 19, 375, 669, July 2, 1973.
- [6] Perez-Santano, A., Trujillano, R.; Belver, C., Gil, A., Vicente, M. A., "Effect of the intercalation condition of montmorillonite with octadecylamine", *Journal of Colloid Interface Science*, 284, 239-244, 2005.